Summer 2017

Inertization, Utilization, and Safe Disposal of Incineration Residues

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INERTIZATION, UTILIZATION, AND SAFE DISPOSAL OF INCINERATION RESIDUES

By

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A Dissertation Submitted to the Faculty of
Old Dominion University in Partial Fulfillment of the
Requirements for the Degree of

DOCTOR OF ENGINEERING
CIVIL AND ENVIRONMENTAL ENGINEERING
OLD DOMION UNIVERSITY
August 2017

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INERTIZATION, UTILIZATION, AND SAFE DISPOSAL OF INCINERATION RESIDUES

Anil Mehrotra
Old Dominion University, 2017
Advisor: Dr. Sandeep Kumar

Combustion of coal or Municipal Solid Waste (MSW) causes air pollution and produces solid residues which contain high levels of toxic elements. The toxic characteristics of residues generated from combustion of MSW in waste-to-energy plants are strictly controlled by Federal and State Waste Management Regulations. According to Resource Conservation and Recovery Act (RCRA), residue generated from combustion of MSW is considered hazardous and must be tested according to EPA Toxic Characteristics Leaching Procedure (TCLP) Method 1311 and suitably treated for its safe disposal to landfills. Experiments with various treatment chemicals as primary independent variable had earlier been conducted by several agencies and facilities. The author has successfully developed two new cost-effective solutions for stabilizing heavy metals in MSW residues to cover the gap between the leachability concentrations of toxic elements observed in residues and the leachability toxicity limits as per EPA's regulatory threshold. These methods include treating MSW residue fly ash (FA) with 2% dolomitic lime by weight, or by injecting aqueous (39% concentration) sodium sulfide at a controlled rate. The extensive full scale experimental study was carried out at 240 t/day capacity Hampton/NASA waste-to-energy mass burn MSW Incinerator (MSWI). This process has showed savings to the extent of $150,000 per year by treating the plant's combustion residues with aqueous sodium sulfide over the use of dolomitic lime for ash treatment.

Results of the prior studies for treatment of toxic wastes have been synthesized and a randomized experimental plan has been planned for conducting this research. Thus valid and defensible results have been obtained that show repeatability of the identified treatment method in varying operating conditions of the combustion process. The research plans and experimental design methods are detailed in section 1.16 of Chapter 1. The treatment method invented has also shown better control of the leachability of toxic heavy metals than previously
used chemical treatment methods. Comparative study showing the level of leachability of toxic heavy metals with different treatment methods are detailed in Chapter 5.

The best management practices for use and disposal of such wastes have been discussed.

Key words: Municipal Solid Waste (MSW); Resource Recovery and Conservation Act (RCRA); Toxic Characteristics Leaching Procedure (TCLP); Combined Ash (CA); Scrubber Dryer Absorber (SDA)
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The contents of this paper are in public domain and can be used without permission of charge.
This thesis is dedicated to my wife Sandhya who always supported and stood by me during all these years of studies and hard work.
ACKNOWLEDGEMENTS

There are many people who have contributed to the successful completion of this dissertation. I extend my many, many thanks to my advisor Dr. Sandeep Kumar and the committee members for their consistent support and guidance during my research. I am also very much thankful to Mr. John McDonald, Manager and all the team members of Hampton/NASA Steam Plant facility for their undaunted support and patience during all the experimental works that I carried out at the facility, faltering in some while succeeding in others, to finally come to a reliable testimony.
**NOMENCLATURE**

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<td>AAS</td>
<td>Atomic Absorption Spectrometry</td>
</tr>
<tr>
<td>ANC</td>
<td>Acid Neutralization Capacity</td>
</tr>
<tr>
<td>APC</td>
<td>Air Pollution Control</td>
</tr>
<tr>
<td>BTU</td>
<td>British thermal unit</td>
</tr>
<tr>
<td>BA</td>
<td>Bottom Ash</td>
</tr>
<tr>
<td>CFR</td>
<td>Code of Federal Regulations</td>
</tr>
<tr>
<td>DEQ</td>
<td>Department of Environmental Quality</td>
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<tr>
<td>EPA</td>
<td>Environmental Protection Agency</td>
</tr>
<tr>
<td>ESP</td>
<td>Electrostatic Precipitator</td>
</tr>
<tr>
<td>FA</td>
<td>Fly ash</td>
</tr>
<tr>
<td>IAWG</td>
<td>International Ash Working Group</td>
</tr>
<tr>
<td>LDR</td>
<td>Land Disposal Restrictions</td>
</tr>
<tr>
<td>LOI</td>
<td>Loss on ignition</td>
</tr>
<tr>
<td>L/S</td>
<td>Liquid-solid ratio</td>
</tr>
<tr>
<td>mg/L</td>
<td>milligrams per liter</td>
</tr>
<tr>
<td>mg/kg</td>
<td>milligrams per kilogram</td>
</tr>
<tr>
<td>mm</td>
<td>millimeter</td>
</tr>
<tr>
<td>MSW</td>
<td>Municipal Solid Waste</td>
</tr>
<tr>
<td>MSWI</td>
<td>Municipal Solid Waste Incinerator</td>
</tr>
<tr>
<td>ppm</td>
<td>part per million</td>
</tr>
<tr>
<td>RCRA</td>
<td>Resource Conservation and Recovery Act</td>
</tr>
<tr>
<td>RDF</td>
<td>Refuse Derived Fuel</td>
</tr>
<tr>
<td>TCLP</td>
<td>Toxicity Characteristic Leaching Procedure</td>
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<tr>
<td>W-t-E</td>
<td>Waste-to-Energy</td>
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CHAPTER 1: INTRODUCTION

1.1 MUNICIPAL SOLID WASTE GENERATION AND UTILIZATION

The garbage generated by households and commercial establishments and managed by local governments is known as municipal solid waste (MSW). MSW is collected and recycled, incinerated, or disposed of in MSW landfills. These types of landfills are generally called sanitary landfills. In the United States the largest component of the MSW stream is paper and card board products (26.6%), with food (14.9%) and yard trimmings (13.3%) the second and third most predominant components (EPA, 2016). Domestic sewage and other municipal wastewater treatment sludges, demolition and construction debris, agricultural and mining residues, and wastes from industrial processes are excluded from the definition of MSW.

Due to substantial increase in populations and consequent increase in generation combustion of MSW and recovery through recycling have increasingly become common MSW management practices worldwide. European Union (EU) countries generate an average of 524 kg of MSW per person per year, while in the US about 730 kg of MSW is generated person/year. In EU27 block 40% of the MSW generated is landfilled, 20% is incinerated, 17% is composted and 23% is recycled. Some northern countries in the EU such as Denmark, Sweden, the Netherlands, and Germany are most advanced in terms of environmental management of their waste and Germany is the foremost among them as less than 5% of the total MSW generated in Germany is landfilled while it recycles 40% of its waste.

Over 250 million tons of MSW is generated in the United States each year, with each citizen generating about 4.4 lbs. of waste per day on an average. Waste recycling including combustion of solid waste that has already been created and collected is considered the best management strategy. Thus the waste is utilized as a secondary raw material and a fuel for production of energy. Incineration of MSW with energy recovery is one the important component of recycling in EPA’s Integrated Solid Waste Management (ISWM) program. According to US EPA’s Advancing Sustainable Materials Management 2014 Fact Sheet, 12.8 % of MSW generated in U.S. is combusted for energy recovery (Figure 1).
EPA implements solid-waste management programs by setting national goals, providing leadership and technical assistance, and developing educational materials. EPA’s Integrated Solid Waste Management (ISWM) program aims at four main components: (1) source reduction and reuse, (2) recycle, (3) energy recovery, and (4) treatment and disposal (EPA, ISWM 2016).

Figure 1 Management of MSW in the United States, 2014

Source: EPA Advancing Sustainable Materials Management: 2014 Fact Sheet

Figure 2 EPA’s Sustainable Waste Management Hierarchy
Source reduction in this hierarchical approach to waste management takes top priority and aims to decrease the volume and toxicity of waste and to increase the useful life of products. As per *EPA Sustainable Materials Management: Non-Hazardous Materials and Waste Management Hierarchy* source reduction can:

- Save natural resources,
- Conserve energy,
- Reduce pollution,
- Reduce the toxicity of our waste, and
- Save money for consumers and businesses alike.

Recycling is the next favored strategy followed by reuse that includes composting and energy recovery through combustion. Landfilling is the least favored option and is to be used for the final disposal of non-recyclables and noncombustible materials. The goal such an integrated management hierarchy is to use a combination of all these methods to handle the MSW stream safely and effectively with the least adverse impact on human health and the environment.

### 1.2 INCINERATION OF MUNICIPAL SOLID WASTE

Incineration of MSW was initially taken up for disposal of residential waste as an alternative to burying it in landfills and the energy released from the combustion of MSW has also been utilized in some form or the other from early times. However, it was during early 1970’s that the incineration of MSW for energy generation was taken up as an organized industry. These facilities came to called Waste-to Energy (W-t-E) or Energy-from-Waste (E-f-W) facilities. Incineration of waste reduces it by about 90% by volume and by about 60-65% by weight. The environmental policies of most of the developed countries call for avoiding disposal in landfills as much as possible.

Worldwide there are presently over 1600 waste-to-energy plants operating at various capacities. One plant currently being built in the Shenzhen megacity of China would be the world’s largest waste-to-energy plant with a capacity to burn 5,000 tons of trash every day. However, much progress in this regard could not be made in the United States which currently
has only 85 such plants in operation. In the US conventional fossil fuels contribute most towards energy generation. Only about 12.8% of the municipal waste generated in the US is used for energy generation while the most of it is still landfilled (Figure 1). According to U.S. Energy Information Administration 67% of the electricity produced in the United States during 2015 was from fossil fuel sources, with coal and natural gas contributing equally, about 33% of it each, and the rest provided by nuclear, wind, hydroelectric and renewables like MSW.

Out of these sources for generation of power, the residues from combustion of coal as well as from MSW incineration contain toxic compounds that create serious environmental hazards. The residue ash from combustion of MSW can leach toxic heavy metals to the ground water if the toxicity is not controlled within permissible limits before its disposal and storage in the landfills.

The coal combustion residues (CCRs) are stored in mono-fills and impoundments and the concentrations of potentially toxic compounds in the coal ash have been determined below the hazardous limits by EPA. But recent accidental spills of CCRs from impoundments in Kingston, Tennessee and the Dan River, North Carolina have raised serious questions about the negative impacts to the ground waters around the impoundments where the coal combustion residues are discharged without any pollution prevention measures. Although about 45% of coal combustion residues generated are recycled for environmentally safer and beneficial applications, the rest 55% are still unsafely stored in impoundments which have the potential to pollute the ground water due to accidental spills and leaching into the surroundings.

Soon after the inception of waste-to-energy facilities public and political concerns were raised regarding the environmental impacts of burning MSW as it produces toxic pollutants that are released to the atmosphere and the residue ashes generated from combustion of MSW contain hazardous heavy metals that have potential to cause groundwater pollution when these residues are landfilled. As a result all countries promulgated progressively higher air emission standards as well as stricter controls on residue ash before its disposal in landfills.

The paper examines various technologies used to control discharge of potentially harmful elements from MSW combustion residues when disposed of in landfills and presents two viable treatment methods as proved by applied research to mitigate the potential negative environmental
impacts MSW incineration (MSWI) residues and provides evidence to the effectiveness of the solutions presented in the context of W-t-E plants operating in the US. The solutions can be applied to reduce the current environmental impacts from the disposal of incineration residues from MSW and can possibly be improved further to deliver better performance. The paper intends to validate the solutions to the practice-based problems in order to deal with the detrimental effects of disposal of incineration residues and is expected to contribute to the body of knowledge in this field.

1.3 UTILIZATION OF MSW AS FUEL

Municipal solid waste is very heterogeneous in characteristics constituting of several organic and inorganic elements and their compounds. Most of the environmental problems of waste disposal are related to the chemicals in the waste. During the incineration process organic components in the waste are oxidized to \( \text{H}_2\text{O}, \ \text{CO}_2, \ \text{NO}_x, \ \text{and CO} \) while the inorganic mineral compounds are either volatilized or remain as solid particles that are trapped in various residue streams. The solid combustion residues in the furnace are collected as bottom ash (BA) which is first quenched in a water trench and then conveyed through an incline conveyor to ash collection area. Before collection the BA is generally passed through a screen to separate oversized unburnt portions and also through a metal separation device – a conveyor passing over a magnet or a rotating magnetic drum picking up ferrous and non-ferrous items in the residue ash.

The volatilized mineral compounds are either discharged to atmosphere with flue gases along with oxidized organics or are sorbed with alkaline sorbent and then condensed out on the fly ash particles collected through particulates collecting devices: Electro Static Precipitators (ESP) or Fabric Filters (FFs) or a combination of both. The prominent sorbent slurry sprayed in the Spray Drier Absorber (SDA) for absorbing acid gases in the flue gas stream is high calcium hydrated lime \( \text{CaOH}_2 \). Other additives like activated carbon and selective non-catalytic reduction (SCNR) agents are used for treatment of dioxins and mercury and for NOx control, respectively. These chemicals along with SDA and ESP/FFs train constitute what is called the Air Pollution Control (APC) device. The dry ash particles collected in the SDA hopper and in the particulate collecting equipment ESP/FFs is called fly ash (FA) which when combined with the courser BA
is collectively called as combined ash (CA). It has generally been found that fly ash contains higher concentrations of toxic inorganic heavy metals than bottom ash.

Figure 3  Management of residue from MSW incineration
Hjelmar, O., 1996

1.4 MSW COMBUSTION TECHNOLOGIES

Two basic technologies are used for incineration of MSW. One is called mass burn (MB) technology which consumes the waste in as-received condition without processing the incoming waste in any manner. The other technology which is also very commonly used is called refuse-derived fuel (RDF) technology in which the refuse is processed in several steps that include breaking open, shredding, screening, and separation of glass and metal etc. Some facilities even use modified RDF technology by densifying the fluffed and fine refuse into briquettes. This technology is called Densified Refuse-derived fuel (DRDF) technology. Each technology has its advantages and disadvantages. The RDF/DRFDF technology increases the heating value of fuel by 25 to 30% but it is very labor and maintenance intensive. The two technologies are discussed in detail in the next sections.

The combined residue from MSW combustion is considered hazardous and must be tested according to EPA Toxic Characteristic Leachability Procedure (TCLP) Method 1311 as
provided in SW-846 guidance manual for meeting the leachability limits of heavy metals into ground water before its safe disposal to sanitary landfills as non-toxic waste or for recycling as secondary material. The EPA TCLP Test Method 1311 is given in Appendix A.

Each ton of municipal solid waste incinerated in a mass burn unit would generate about 2% to 4% (40 – 80 lb.) of hazardous waste. The residues collected in APC system include the particulate matter captured after the acid gas treatment device, this waste can either be solid or liquid slurry depending on the type of air pollution control equipment used which may be dry, semi-dry or a wet process.

![Figure 4](source: https://www.epa.gov/sites/production/files/2015-06/documents/basic_information_about_energy_recovery_from_waste_web_pdf.pdf)  

**Figure 4** Schematic of a mass burn MSW incineration process

*Source: Basic Information about Energy Recovery from Waste, EPA Archives*

### 1.5 MASS BURN COMBUSTION TECHNOLOGY

Mass burn (MB) is the dominant waste-to-energy technology in which MSW is combusted on moving grates in “as-received” condition. It is the simplest technology that has been in use for several decades. The MSW is combusted as-received without any pre-processing
of fuel; only very large and hazardous objects are pulled out from the refuse pile. In large mass-burn facilities refuse up to 150 tons per hour is fed into the hoppers. The refuse moves down the feed hopper by gravity and is then pushed into the furnace by heavy-duty feed rams that are hydraulically operated. The fuel is processed through 2 or 3 sections of moving stokers that are set at a gradient. The process takes about an hour and quite a high degree of combustion is achieved. Primary combustion air is injected through the grates and tuyers and the secondary air flows through nozzles above the grates to help in combustion of unburnt carbon in the flue gases before they exit the furnace.

The technology has now attained a high degree of development. Good combustion practice and state-of-the-art dedicated digital controls (DDCs) have resulted in higher rate of capture or destruction of pollutants, like sulfur, chlorine, carbon mono oxide, dioxins, furans, volatile metals, and particulate matter.

1.6 RDF COMBUSTION TECHNOLOGY

Refuse-Derived-Fuel (RDF) technology is a simple advancement over the MB process. The refuse is shredded, crushed in hammer mill, and screened through trommel into a less heterogeneous fuel which is subsequently subjected to separation and recycling of unburnable materials, like metal and glass. The easily accessible recyclable materials are manually picked up from slow-moving conveyors, while some ferrous metals are later recovered through magnetic rotating drums and non-ferrous metals are captured by eddy-current separators. The pre-processing of municipal solid waste increases the caloric value of the fuel and hence the capacity of the combustion units. While average higher heating value (HHV) of “as-received” MSW used in MB process is 4,500 BTU/lb, pre-processing of solid waste as refuse-derived-fuel (RDF) increases the HHV of MSW by about 25% to approximately 6,500 BTU/lb.
In preparing RDF the pre-processing of MSW is carried out in several steps as shown the flow diagram below.
There are, however, several disadvantages of RDF technology. Major problems are encountered when explosive objects like propane gas cylinders go undetected through the incoming solid wastes and cause explosions when processed through giant hammer rotated at high speeds. Shredders and hammer mills are now equipped with explosion-containment devices above their chambers but sometimes explosions put the equipment out of order for long periods of time requiring extensive maintenance.

Waste-to-Energy Research Technology Council (WTERT), Earth Engineering Center, Columbia University developed a new generation of high-torque, low-speed shredders equipped with mechanisms to detect and discard large and metallic objects in order to avoid this type of catastrophic problems. The technology has been used in newer RDF plants like South East Massachusetts (SEMASS) facility (NAWTEC, 2000). Because RDF process is equipped with a series of pre-processing equipment mentioned and with multiple set of conveyors, the process becomes prone to breakdowns and hence is very labor and maintenance-intensive. About twice the size of labor force is needed to operate an RDF plant than that for a MB plant.

When examined from the point of view of reaction kinetics, when the highly heterogeneous MSW is shredded to smaller uniform size during pre-processing in RDF plants, its heat and mass transfer rates are increased. The homogenized fuel allows for easier access of primary air from underneath the stoker grates thus increasing the drying, volatilizing, and higher combustion rates in the RDF furnace. The secondary combustion occurring in suspension is also higher than in MB system.

A study of the design of an RDF plant operated by South East Massachusetts (SEMASS) utilizing RDF technology and two mass burn units Union County Stoker WTE and Brescia Stoker WTE was conducted by Earth Engineering Center, Columbia University. Both these plants are operated by Covanta Energy. The study was conducted to determine the difference in rates of combustion per unit surface area of grates between the two types of technologies based on the respective physical dimensions, MSW feed rates, and air injected in these plants. The results of the study are shown in the Table 1.
Table 1  MB and RDF WTE COMBUSTION PLANT DESIGNS

SEMASS: South Eastern Massachusetts (RDF-type plant of COVANTA)


<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Capacity, tons/day (per unit)</td>
<td>480</td>
<td>792</td>
<td>910</td>
</tr>
<tr>
<td>Heating value of fuel, MJ/kg</td>
<td>11</td>
<td>11.3</td>
<td>11.63</td>
</tr>
<tr>
<td>Process gas volume, Nm³/hour</td>
<td>125,300</td>
<td>135,000</td>
<td>208,500</td>
</tr>
<tr>
<td>Process gas volume/ton, dry Nm³</td>
<td>5,653</td>
<td>4,100</td>
<td>5,500</td>
</tr>
<tr>
<td>Length of grate, m</td>
<td>7.5</td>
<td>8</td>
<td>6</td>
</tr>
<tr>
<td>Width of grate &amp; furnace, m</td>
<td>7.8</td>
<td>12.8</td>
<td>10</td>
</tr>
<tr>
<td>Grate area, m²</td>
<td>58.5</td>
<td>102.4</td>
<td>60</td>
</tr>
<tr>
<td>Grate productivity, tons/day/m²</td>
<td>8.2</td>
<td>7.7</td>
<td>15.2</td>
</tr>
<tr>
<td>Heat generation rate, MW (Thermal)</td>
<td>55.5</td>
<td>94.2</td>
<td>11.4</td>
</tr>
<tr>
<td>Heat flux released on grate, MW/m</td>
<td>0.95</td>
<td>0.92</td>
<td>1.86</td>
</tr>
<tr>
<td>Length of furnace, m</td>
<td>6.5</td>
<td>5</td>
<td>6</td>
</tr>
<tr>
<td>Furnace cross section, m²</td>
<td>51</td>
<td>64</td>
<td>60</td>
</tr>
<tr>
<td>Velocity of gas in combustion chamber, m/s</td>
<td>2.7</td>
<td>2.3</td>
<td>3.8</td>
</tr>
<tr>
<td>Reynolds number in furnace (@ 900°C)</td>
<td>100,000</td>
<td>66,000</td>
<td>130,000</td>
</tr>
<tr>
<td>Furnace height, m</td>
<td>19</td>
<td>22</td>
<td>30</td>
</tr>
<tr>
<td>Average gas residence time, s</td>
<td>7.0</td>
<td>9.5</td>
<td>7.9</td>
</tr>
<tr>
<td>Waterwall surface area, m²</td>
<td>543</td>
<td>783</td>
<td>960</td>
</tr>
<tr>
<td>Heat flux at waterwall (50% load), MW/m²</td>
<td>0.05</td>
<td>0.06</td>
<td>0.06</td>
</tr>
</tbody>
</table>

The study indicated that the grate productivity in terms of tons/day/m² of RDF plant was 83% higher than the Union County MB plant in USA, and it was 96% higher as compared to the
Brescia Stoker MB WTE facility in Italy. Grate productivity is measured in terms of tons of MSW processed/day/unit grate area (m²) as given in above study. Higher grate productivity in RDF plant was expected due to higher rate of combustion because of pre-shedding of the refuse and more efficient furnace design.

1.7 CHARACTERIZATION OF MSW RESIDUES

The MSW incineration residue characteristically contains high concentrations of salts, heavy metals, and organic trace pollutants. Typical concentrations of heavy metals in the bottom ash portion of residue ashes generated in a Municipal Solid Waste Incinerator (MSWI) are shown in the following table (Journal of Hazardous Materials 47, 1996).

<table>
<thead>
<tr>
<th>Heavy metal</th>
<th>Range for bottom ash</th>
<th>Range for fly ash</th>
<th>Range for dry/semidry APC system residues</th>
<th>Range for wet APC system residue without fly ash</th>
</tr>
</thead>
<tbody>
<tr>
<td>As</td>
<td>0.12-190</td>
<td>37-320</td>
<td>18-530</td>
<td>41-210</td>
</tr>
<tr>
<td>Ba</td>
<td>400-3000</td>
<td>330-3100</td>
<td>51-14000</td>
<td>55-1600</td>
</tr>
<tr>
<td>Cd</td>
<td>0.30-71</td>
<td>50-450</td>
<td>140-300</td>
<td>150-1400</td>
</tr>
<tr>
<td>Cr</td>
<td>23-3200</td>
<td>140-1100</td>
<td>73-570</td>
<td>80-560</td>
</tr>
<tr>
<td>Hg</td>
<td>0.02-7.80</td>
<td>0.70-30</td>
<td>0.10-51</td>
<td>2.20-2300</td>
</tr>
<tr>
<td>Pb</td>
<td>98-14000</td>
<td>5300-26000</td>
<td>2500-10000</td>
<td>3300-22000</td>
</tr>
<tr>
<td>Se</td>
<td>0.05-10</td>
<td>0.40-31</td>
<td>0.70-29</td>
<td>-</td>
</tr>
<tr>
<td>Si</td>
<td>91000-33000</td>
<td>95000-210000</td>
<td>36000-120000</td>
<td>78000</td>
</tr>
</tbody>
</table>

All concentrations are in mg/Kg

1.8 COMPARISONS OF MASS BURN AND RDF ASH CHARACTERISTICS

As indicated in Section 2.2 RDF Combustion Technology, the grate productivity in terms of tons/day/m² of RDF plants is greater than that of plants that are constructed with mass burn systems. The better combustion rate of RDF systems results in higher productivity and also results in lower CO₂ emissions and thus in lower pollution of the environment from greenhouse gases (GHGs).

However, due to higher energy required to process raw MSW into RDF the overall system efficiency of RDF plants is lower than that of MB plants. As per the system used by EPA to work out the combustion system efficiency from conversion of MSW to energy (most of the WTE plants in the United States produce electricity) the total system efficiency has been estimated as 17.8% for MB and 16.3% for RDF (US EPA, Combustion). These data are provided in Appendix A.

The bottom ash from combustion of RDF, which is more homogeneous and less coarse than “as-received” raw MSW, has found some possibilities for its utilization in road paving and mixed with other materials for cement production.

1.9 CHEMICAL COMPOSITIONS OF ASH FROM MASS-BURN AND RDF SYSTEMS

A study presented during North American Waste-to-Energy Conference (NAWTEC) in 1997 indicated chemical composition of residue ash from the two main MSWI technologies, mass burn and RDF, are shown in the Table 3.
Table 3  Comparisons of chemical compositions (wt%) of ash from mass burn (MSW) and RDF

<table>
<thead>
<tr>
<th>Inorganic Oxides</th>
<th>MB</th>
<th>RDF</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Bottom ash (%)</td>
<td>Fly ash (%)</td>
</tr>
<tr>
<td>CaO</td>
<td>34.678</td>
<td>16.901</td>
</tr>
<tr>
<td>SiO₂</td>
<td>18.653</td>
<td>12.481</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>13.973</td>
<td>5.946</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>27.053</td>
<td>48.341</td>
</tr>
<tr>
<td>ZnO</td>
<td>-</td>
<td>13.336</td>
</tr>
<tr>
<td>MgO</td>
<td>5.492</td>
<td>-</td>
</tr>
<tr>
<td>Cr₂O₃</td>
<td>-</td>
<td>2.926</td>
</tr>
</tbody>
</table>


1.10  HEAVY METALS IN RESIDUES FROM MASS-BURN AND RDF COMBUSTION

According to the same study by NAWTEC the Toxic Characteristic Leaching Procedure (TCLP) analysis of heavy metals in bottom ash and fly ash from MB MSW and RDF has indicated that although the concentration of Pb falls below the TCLP standards, leaching of Cd remains higher than TCLP standards for residue ash from both MB MSW and RDF (NAWTEC, 1997). It was inferred that the BA generated from burning MSW in “as-received” condition and as RDF can be classified as non-hazardous, but both types of fly ash are required to be treated due to higher contents of toxic metals. The results are shown in the table below.
Table 4  TCLP analysis of bottom and fly ash from combustion of MB and RDF

<table>
<thead>
<tr>
<th>Toxic metals/pH</th>
<th>Mass Burn MSW</th>
<th>RDF</th>
<th>TCLP Standards</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Bottom ash</td>
<td>Fly ash</td>
<td>Bottom ash</td>
</tr>
<tr>
<td>As (mg/L)</td>
<td>ND &lt; 0.001</td>
<td>ND &lt; 0.001</td>
<td>ND &lt; 0.001</td>
</tr>
<tr>
<td>Cd (mg/L)</td>
<td>0.01 – 0.02</td>
<td>4.60 – 4.67</td>
<td>0.05 – 0.06</td>
</tr>
<tr>
<td>Cu (mg/L)</td>
<td>0.03 – 0.40</td>
<td>22.30 – 22.40</td>
<td>0.39 – 0.40</td>
</tr>
<tr>
<td>Cr (mg/L)</td>
<td>0.03 - 0.04</td>
<td>ND &lt; 0.02</td>
<td>0.12 - 0.13</td>
</tr>
<tr>
<td>Hg (mg/L)</td>
<td>ND &lt; 0.002</td>
<td>ND &lt; 0.002</td>
<td>ND &lt; 0.002</td>
</tr>
<tr>
<td>Pb (mg/L)</td>
<td>ND &lt; 0.03</td>
<td>9.48 – 9.65</td>
<td>0.11 – 0.12</td>
</tr>
<tr>
<td>Zn (mg/L)</td>
<td>1.50 – 1.60</td>
<td>5.22 – 5.34</td>
<td>16.10 – 16.30</td>
</tr>
<tr>
<td>pH</td>
<td>11.8</td>
<td>5.6</td>
<td>10.2</td>
</tr>
</tbody>
</table>

As shown in Table 4 all 7 toxic metals extracted from the bottom ash of MB and burning RDF exhibit relatively lower concentrations as compared to fly ash. The extracted metals from the fly ash in the RDF incineration process generally exhibit relatively lower concentrations than that of MB, still these concentrations are higher than the regulatory limits and therefore the ashes are classified as hazardous materials. The extractable cadmium concentrations are beyond the regulatory levels in both MB and RDF plants. The substantial differences require the fly ash or combined ash, if the ash generator disposal program includes other streams of ashes, from both combustion technologies to be treated by either solidification, stabilization, evaporation or vitrification techniques that are discussed later in this paper.

Some kind of pre-treatment is therefore inevitably required for both types of MSW incinerators in order to improve their environmental characteristics and possibilities of reuse. Various treatment methods used are discussed in this paper. These can be broadly categorized as separation process, solidification and stabilization by additives or use of chemicals, and thermal methods.
1.11 STUDY SITE FOR THIS RESEARCH

The WTE facility Hampton/NASA Steam Plant located at NASA Langley Research Center in Hampton, Virginia has been chosen for purpose of studying the treatment methods for plants utilizing MSW as combustion fuel for generation of steam and electricity. The facility’s letter authorizing the use of data from various tests and methods used for control of leachability of heavy metals in its residue ash is attached at Appendix C.

This facility has been in operation since 1980. It operates two municipal waste combustors, each combusting 120 tons per day (total 240 tons/day) of MSW to recover steam energy for supply to nearby NASA center.

Municipal waste combustors (MWCs) that feed 250 tons or less of MSW per day are classified as Class II facilities according to EPA municipal solid waste incineration (MSWI) classifications. The MSW combustion residues are considered hazardous as EPA’s Resource Conservation and Recovery Act (RCRA) Land Disposal Restrictions (LDR) and as covered by the Code for Federal regulations 40CFR 261 governing all hazardous wastes. Accordingly the MSW combustion residues have to be tested for the following RCRA Subpart C –Characteristics of Hazardous Waste before their reuse or disposal to landfill:

1. § 40CFR 261. 21 Characteristic of Ignitability
2. § 40CFR 261. 22 Characteristic of Corrosivity
3. § 40CFR 261. 23 Characteristic of Reactivity, and
4. § 40CFR 261. 24 Toxicity characteristic

The toxicity of the MSW residue is tested by EPA TCLP Method 1311. The residue ash generated at Hampton/NASA Steam Plant has always met with the TCLP regulatory limits without requiring any treatment of its combustion residue until it modified its Air Pollution Control (APC) system during late 2005 to meet EPA’s new air emission guidelines (EG).

The Hampton/NASA Steam Plant follows a standardized procedure for collection of a random representative residue ash sample for TCLP testing. The procedure is included in Appendix B of this paper.
Most of the combustion process residue is BA which is quenched in a wet bottom trench and then conveyed through an incline conveyor to the vibrating screen. Ferrous metal is removed by a rotating drum magnet and the scrubber ash and APC system fly ash is mixed with the bottom ash after conditioning with boiler process water. Before mixing with bottom ash the finer fly ash is subjected to chemical treatment. Initial treatment chemical used during early 2006 was a proprietary product. An alternative chemical dolomitic lime was later used starting in November 2008.

A snapshot (Figure 7) of the flue gas cleaning (in scrubber) and fabric filter particulate collection system is shown below. The sorbent of choice for flue gas scrubbing to remove sulfur dioxide and HCL (a hazardous air pollutant) is high calcium hydrated lime. This is followed by a set of particulate collection equipment, for example fabric filters in case of Hampton plant.

![Figure 7 Hampton/NASA Steam Plant modified Air Pollution Control System](image)

Most of the lead, cadmium and other TCLP metals leave the boilers with flue gas and are condensed in scrubber and then captured in the fabric filters in air pollution control residues. These fine dry residues are conveyed through a set of enclosed conveyors at gradient to mix
these residues with the coarser bottom ash. Before mixing these residues are treated with heavy metal stabilizing chemical and conditioned with hot boiler process water.

A snap shot of scrubber and fly ash conveying system and its treatment is shown in Figure 8.

Figure 8  Scrubber and fly ash conveying system residue ash and its treatment
1.12  MASS FLOWS IN GRATE OPERATED MB INCINERATION PLANTS

Grate furnaces, mostly reciprocating type, are generally a preferred option with waste incineration because of their ability to handle high feed inputs. These grates are able to feed untreated as-received MSW of any particle size and shape. The air emissions and combustion residues from grate furnaces are distributed into various fractions. These fractions lie in certain range and show some variations depending on the type of air pollution control (APC) system used and on the feeding capacities of different types of MSW incinerators, but still broadly follow a set pattern.

The air emissions from state-of-the-art MSW plants normally constitute 68 - 70% of various gases, 24 - 26 % moisture, and about 5% solid particles of various metal compounds and aerosols.

The bottom residues are divided in the following fractions as percentages of refuse feed:

Table 5  Normal percent fractions of MSW combustion residue

<table>
<thead>
<tr>
<th>Constituent of bottom residue</th>
<th>% of refuse feed</th>
<th>% of total ash</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Furnace bottom ash (BA) including grate siftings</td>
<td>27.0%</td>
<td>80.1%</td>
</tr>
<tr>
<td>2. Scrubber ash and Fabric Filter ash: Fly ash (FA)</td>
<td>3.3%</td>
<td>9.8%</td>
</tr>
<tr>
<td>Sub-total of combined ash (BA + FA = CA)</td>
<td></td>
<td>89.9%</td>
</tr>
<tr>
<td>3. Waste water</td>
<td>2.0%</td>
<td>5.9%</td>
</tr>
<tr>
<td>4. Scrap metal (post combustion separation)</td>
<td>1.4%</td>
<td>4.2%</td>
</tr>
<tr>
<td>Total</td>
<td>33.7%</td>
<td>100.0%</td>
</tr>
</tbody>
</table>

The above fractions have been arrived at based on the studies as the one shown in the figure below and it matches with the generally accepted fact that the refuse feed when combusted...
in a municipal solid waste incinerator (MSWI) is reduced to about 10 - 12% in volume and to about one third (33%) in weight (Vehlow, 2012).

Figure 9 Fractions of MSW Incineration residues per 1000 kg refuse feed


As discussed above in this paper Hampton/NASA Steam Plant which is the site chosen for this study operates 2 boilers each with a refuse feed capacity of 120 tons per day. The breakup of various fractions of residue generated from the total refuse feed rate of 240 tpd from the two boilers is worked out as given in the table below.

Table 6 Fractions of MSW combustion residue: Hampton Steam Plant

<table>
<thead>
<tr>
<th>Constituent of bottom residue</th>
<th>% of refuse feed</th>
<th>Fraction (tpd), of 240 tpd feed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Furnace bottom ash (BA) including grate siftings</td>
<td>27.0%</td>
<td>64.80 tons</td>
</tr>
<tr>
<td>2. Scrubber ash and Fabric Filter ash: Fly ash (FA)</td>
<td>3.3%</td>
<td>7.92 tons</td>
</tr>
<tr>
<td>Sub-total of combined ash (BA + FA = CA)</td>
<td></td>
<td>72.7 tons</td>
</tr>
<tr>
<td>3. Waste water</td>
<td>2.0%</td>
<td>4.80 tons</td>
</tr>
<tr>
<td>4. Scrap metal (post combustion separation)</td>
<td>1.4%</td>
<td>3.36 tons</td>
</tr>
<tr>
<td>Total</td>
<td>33.7%</td>
<td>80.88 tpd</td>
</tr>
</tbody>
</table>
It is thus calculated that about 72 tons of combined ash (CA) is the amount of total ash that is generated each day by operating two boilers each with 120 tpd refuse feed capacity, and that is the total ash that needs to be treated for solidification/stabilization of toxic elements so that these are immobilized and their leaching within the regulatory limits when the ash is disposed of in the landfill.

1.13 PROBLEM STATEMENT

Advancements during the last two decades in the state-of-the-art modern MSWI technologies and air pollution control (APC) measures have considerably shifted the constituents of concern (toxic elements) from air emissions from these MSWIs to their combustion residues. These residues when either reused as building or road construction materials or disposed of in landfills have the potential to leach toxic pollutants in soil and water.

Table 4 gives the TCLP Standards for toxicity limits of heavy metals in MSW residue ashes.

The APC system modification at the Hampton/NASA Steam plant during November 2005 changed the kinetics of residue ash, especially the fly ash collected from the combustion flue gases, and the combined residue ash including the furnace BA when tested by TCLP Method 1311 was found to have leachability of heavy metals, mainly Cadmium and Lead beyond the EPA regulatory limits.

Leachability of heavy metals especially Cd showed in excess of regulatory threshold when tested as per TCLP method after APC modifications were completed. Table 7 gives some results when Cd in residue ash first tested over the regulatory limits after APC system at Hampton/NASA Steam Plant was upgraded in November, 2005. The concentrations of Cd were found beyond the regulatory threshold.
Table 7  Initial gaps in leachability results and EPA limits

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Date</th>
<th>Results (mg/L)</th>
<th>EPA Threshold</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Cd  Pb</td>
<td>Cd: 1.0 mg/L</td>
</tr>
<tr>
<td>HSP-0206-6A</td>
<td>2/3/2006</td>
<td>1.17 0.221</td>
<td>Pb: 5.0 mg/L</td>
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<tr>
<td>HSP-0206-8A</td>
<td>2/4/2006</td>
<td>1.55 0.293</td>
<td></td>
</tr>
<tr>
<td>HSP-0206-13A</td>
<td>2/13/2006</td>
<td>1.38 0.114</td>
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</tr>
<tr>
<td>HSP-0406-C1</td>
<td>4/8/206</td>
<td>0.929 0.822</td>
<td></td>
</tr>
<tr>
<td>HSP-0406-C2</td>
<td>4/11/206</td>
<td>1.55 22.2</td>
<td></td>
</tr>
<tr>
<td>HSP-0406-C2A</td>
<td>4/11/206</td>
<td>1.95 10.4</td>
<td></td>
</tr>
<tr>
<td>HSP-0406-C3</td>
<td>4/13/206</td>
<td>1.35 7.11</td>
<td></td>
</tr>
<tr>
<td>HSP-0406-C4</td>
<td>4/17/206</td>
<td>1.92 14.2</td>
<td></td>
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<tr>
<td>HSP-0406-C4A</td>
<td>4/17/206</td>
<td>1.51 12.5</td>
<td></td>
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<tr>
<td>HSP-0406-C5A</td>
<td>4/19/206</td>
<td>1.64 9.02</td>
<td></td>
</tr>
</tbody>
</table>

*Source:* Hampton Steam Plant data

To overcome the gap between the EPA’s leachability limits for heavy metals and the values obtained in the facility’s residue ash it became necessary to apply some chemical treatment for stabilization of heavy metals to make it non-hazardous before disposal to sanitary landfill. This was achieved by first using a proprietary chemical and later with dolomitic lime.

The facility further considered following options in this regard:

i) Construct a storage silo large enough to store long-term supplies of dolomite lime transported in bulk trucks to avoid paying heavily for supply in super sacks.

ii) Make process/chemical use changes upstream of fly ash generation, for example to increase spraying of high calcium hydrated lime (which is stored in a silo and mixed with water to make slurry) or to spray a mix of high calcium hydrated lime and dolomitic lime in the flue gases to ascertain if it will change the reaction kinetics to the extent that may help eliminate use of dolomitic lime in the fly ash collection system downstream of the flue gas path.
iii) Find an alternative to dolomite lime in form of a liquid chemical injection that would use an existing process water injection as part of fly ash conditioning. A small liquid storage tank and pump will be needed for this system in case it is determined to treat the ashes.

iv) Trials with sodium sulfide liquid chemical injection as part of proposal in (iii) above were undertaken and the results obtained are discussed.

1.14 PURPOSE OF THE STUDY

The study outlines and scrutinizes the effectiveness of various fly ash chemical treatment methods currently available to stabilize and immobilize heavy metals in the combined ash that is generated through combustion of MSW in waste-to-energy plants. It applies the results to find a more cost-effective method of treatment of combustion ash beyond those that have been used so far at the Hampton/NASA Steam Plant waste-to-energy facility that has been selected for this study.

The purpose of the study is to establish a treatment method for fly ash to control the concentrations Cd and Pb in the combined ash so that when tested for leachability the concentrations of these metals remain within the EPA regulatory limit of 1 mg/L for Cd and 5 mg/L for Pb so that waste is classified as non-hazardous and safe for disposal in sanitary landfills. As a further goal of the study is to optimize the quantitative and qualitative injection of identified chemical treatment and process controls in the fly ash downstream system in accordance with the variations in the mass flux rate of generation of residue wastes due to the upstream process variations in the operational status of either one or both of the boilers.

1.15 STUDY METHOD

Data for heavy metals concentrations from analytical testing of residue ash Waste-to-Energy Hampton/NASA Steam Plant during past several years are studied. The goal of the research is to develop cost-effective solutions to cover the gap between the leachability concentrations of toxic elements observed in residues from thermal conversion processes of MSW and other solid fuels for energy recovery and the leachability toxicity limits as per EPA’s
regulatory threshold. The study explores the best management practices for use and disposal of such wastes. Experimental data are generated by developing and employing process controls and alternative treatment methods and compared with EPA regulatory limits for leaching of heavy metals.

1.16 RESEARCH PLAN AND EXPERIMENTAL DESIGN METHODS

A detailed research plan was worked out and design methods were adopted to represent field conditions while conducting experimental research.

1.16.1 RESEARCH PLAN

The integrating dimensions of the project are based on multidisciplinary design optimization using experimental methodologies decomposed in following steps:

i) Defining clearly the domain of the research project
ii) Identifying set of prior studies that met the priori criteria regarding the phenomenon in question
iii) Synthesizing prior research and conducting valid, defensible literature reviews meeting a strong scientific rigor as applied in the data analyses
iv) Developing a randomized experimental design meeting internal validity criteria
v) Conducting experiments at Hampton Steam Plant and estimating causal effects of treatments in random studies
vi) Carrying out initial Exploratory Data Analysis (EDA) for analyzing data from experiments in order to meet the following procedural steps:
   • Detection of mistakes
   • Checking of assumptions
   • Preliminary selection of appropriate models
   • Determining relationships among the explanatory variables, and
   • Assessing the direction and rough size of relationships between explanatory and outcome variables.
vii) Analyzing results by applying parametric inferential statistics and regression techniques

viii) Assessing repeatability of results that satisfy decision criteria and meeting the dimensions of their reproducibility for the entire population at the selected confidence interval of 90%, one-tailed

ix) Integration, validation, and qualification of results

x) Reporting project results and limitations

The study has adopted a quantitative experimental design approach with identified independent and dependent variables for different types of controls and treatment methods to study their cause and effect. It incorporates measures as enumerated above and as appropriate in conduct of this research.

The results from successive use of different treatment methods as listed below and adopted sequentially at various intervals are discussed in this report:

1. Treatment method with a proprietary technology

2. Switch over to cost-effective dolomitic lime fines

3. Use of Dolomitic Hydrated Lime to replace high calcium hydrated lime for flue gas scrubbing

4. Use of increase concentration of High Calcium Hydrated lime with parametric changes in Flue Gas scrubbing conditions

5. Eliminate use of dolomite taking advantage of alkalinity of boiler process water used for conditioning of fly ash

6. Injecting sodium sulfide Na₂S 39% aqueous solution in fly ash conditioning system

1.16.2 EXPERIMENTAL DESIGN APPROACH

The applied research requires the collection and interpretation of data and is based on the systems engineering V-process: the problem of finding the well-performing solution for the treatment of incineration residues has been worked out within the environmental, technological,
and economic constraints by breaking up the problem into more manageable sub-problems and then systematically synthesizing the various solutions.

The suggested solution is then examined by verification and validation through qualified test methods and by performing a scientifically determined number of tests to prove its efficacy.

The process development has thus followed the applied systems engineering V-process as depicted below- defining and breaking up the problem on the left and then integrating and qualifying the solution on the right of the V-process (Buede, D. M. 1999, 10).

Where, CI mentioned in the text boxes stands for Configuration Integration.

Figure 10  Systems Engineering “Vee”, Engineering Design of Systems, Dennis M. Buede, 1999

1.16.3 EXPERIMENTAL DESIGN METHODS

The goal of the experimental design method (Figure 12) was to make correct and objective inference about the process adopted to control the leachability of toxic heavy metals in
municipal solid waste incineration residues within regulatory threshold based on information collected from the experiment.

The results of the experiment were then planned to be used to characterize the system and verify if the outcome or solution can be reproduced for use in similar systems and it is capable of being used at any scale of operations.

Figure 11  Experimental design methods

Following three basic principles were adopted in designing the experiments:

1. **Replication**
   It was aimed that the results of the experiment can be applied in similar incineration
processes by reducing the effects of minor deviations (noises) in original variables.

2. **Randomization**
   It was meant to balance out any internal/external influence from the “ill-behaving” variables towards the target solution.

3. **Using blocks (process variations)**
   This was used to cover various categories of process changes and to ensure that the target solution would be effective in usual applicable process conditions.

Keeping the above fundamental principles in mind the experimental design process was carried out in the following steps:

1. **Problem conceptualization**
   Recognition and statement of the problem

2. **Choice of factors**
   Treatment as primary control and blocks (process running conditions) as secondary control

3. **Selection of response variables**
   Variables that might affect the results of the experiments were selected

4. **Choice of Experimental Design**
   Randomized Complete Block (RCB) design was chosen as this design approach is very flexible for use in any number of treatments and any number of blocks.

5. **Performing Experiments and Collecting Samples**
   EPA guidelines for sampling procedures according to Method 1311 were used

6. **Analysis, conclusions and recommendations**
   Samples were sent for analysis to a certified chemical laboratory under an established chain of custody procedure, and were analyzed as EPA Method 1311.
   Results of the concentrations of toxic elements obtained as per TCLP tests were statistically analyzed, concluded as findings of the research, and used for recommendations.

Following calculation steps are used in the Randomized Complete Block (RCB) design:

\[ y_{ij} = \mu + \tau_i + \beta_j + \varepsilon_{ij} \quad (i = 1, \ldots, \text{treatments}; \quad j = 1, \ldots, \text{blocks}) \]

Where,
\[ y_{ij} = \text{Response on (i, j)th observations} \]
\[ \mu = \text{Overall mean} \]
\[ \tau_i = i\text{th treatment effect} \]
\[ \beta_j = j\text{th block effect} \]
\[ \varepsilon_{ij} = \text{Random error due to (i, j) th Obs. Where } \varepsilon \sim NID(0, \sigma^2) \]

**F-test based Test of Hypothesis (T.H.) at given level of confidence**

a) Test of **treatment** effects

\[ H_0: \tau_1 = \tau_2 = \cdots = \tau_t = 0 \]
\[ H: \text{At least one } \tau_i \neq 0 \]

t.s. \[ F_0 = \frac{\text{MS(Treatment)}}{\text{MSE}} \]

b) Test of **block** effects

\[ H_0: \beta_1 = \beta_2 = \cdots = \beta_b = 0 \]
\[ H: \text{At least one } \beta_i \neq 0 \]

t.s. \[ F_0 = \frac{\text{MS(Block)}}{\text{MSE}} \]

These calculation steps are used later under Chapter 6 Discussions and Statistical Analysis.
CHAPTER 2: RESIDUE ASH TESTING PROCEDURES

2.1 DESCRIPTION OF RESIDUE ASH TESTING PROCEDURES

During the course of developing MSW residue ash test procedures to determine the Toxicity of leachate when disposed of in landfills EPA initially used extraction procedure (EP) test that was modified as Modified Waste Extraction Procedure (MWEP) or water batch test using distilled or ionized water for extraction.

EPA then designed Toxic Characteristics Leaching Procedure (TCLP) test to simulate wastes sitting inside the landfills for a number of years to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphase wastes in landfills. TCLP test procedure methods are detailed in municipal solid waste manual SW-846 Method 1311.

EPA also developed a Synthetic Precipitation Leaching Procedure (SPLP) test. Details of this test procedure methods are provided in municipal solid waste manual SW-846 Method 1312. SPLP was designed to simulate waste material sitting in-situ, i.e. in or on top of the ground surface. Results from SPLP tests are utilized to develop site-specific soil remediation criteria that will be protective of groundwater from excessive contamination from leachate. The primary difference between SPLP and TCLP is the use of different extraction fluids which are dictated by what each test is designed to simulate.

Another test method used is sequential or multiple extraction procedure (MEP) with details of the procedure covered in SW-846 Method 1320.

2.2 TCLP TEST BY EPA METHOD 1311

US EPA has chosen TCLP Method 1311 for testing concentrations of heavy metals. The TCLP procedure uses statistical population Upper Confidence Level 90% (UCL90) one-sided limits (Sample Analysis Guidance Document SW-846). The details of the method are given in Appendix A.
The test uses acetic acid solution to “force” leaching and maintain a prescribed pH to rapidly extract the metals from ash extracts while simulating worst case scenarios of ash disposal. These procedures are designed to provide data artificially in the absence of actual field leachate data to simulate ash leachate characteristics. The TCLP procedure consists of single batch 18-hour simulation at pH = 4.93 for ash pH < 5 (called TCLP Fluid 1) or pH = 2.88 for ash pH > 5 (TCLP Fluid 2). MWC residue ash generally has a pH > 10. The extractions are run under conditions of low (acidic) pH to mimic conditions typically found in landfills containing decomposing organic matter.

Data obtained from TCLP test are used to determine whether a solid waste (residue ash) exhibits the hazardous waste characteristics of toxicity. Solids that fail the TCLP are considered to be hazardous waste under RCRA and cannot be disposed of in landfills. In such case the residue ash is either required to be treated to stabilize or immobilize the heavy metals from leaching or otherwise the waste is to be discarded in separate hazardous waste disposal sites. Solid wastes subjected to TCLP are considered to exhibit Toxic Characteristic (TC) if the waste sample leaches a TC constituent at a level equal to or exceeding the regulatory limit set forth in 40 CFR 261.24, as per TCLP Standards given in Table 4.

### 2.3 STATISTICAL METHOD FOR ANALYSIS OF TCLP TEST RESULTS

Following data evaluation approach is adopted in accordance with EPA SW-846 Test Methods for Evaluating Solid Wastes:

1. Determine the mean concentration ($\bar{x}$) of the 8-hour composite samples.
2. Determine the standard deviation ($s$) of the data employed to calculate the mean (i.e., the individual composite extract results)
3. Determine the upper limit at a 90% level of confidence (one-tailed) for the mean of each analyte.
4. If the 90% level of confidence (one-tailed) is less than the applicable Regulatory Threshold (RT) as listed in the Table 7 above, then the waste (ash) passes the TC.
5. Results from the multiple events for the same waste can be combined (pooled) into one data set, and a new confidence interval calculated if the sampling and laboratory analysis were the same for all sampling and analysis events.
6. Use Student’s t-test method to compare population means if the underlying population has a normal distribution, otherwise use the Wilcoxon rank Sum Test (also known as the Mann-Whitney U Test) to test whether the populations are identical but not normal.

7. Reasons for “outliers”, if any, should be determined, which may include:
   - Contaminated sample equipment
   - Laboratory contamination of the sample
   - Errors in transcription of the data values

   Once a specific reason is documented, the result should be excluded from any further statistical analysis.

2.4 SAMPLING PROCEDURE

Sample collection and preparation for TCLP tests is carried out in the following manner.

In order to ensure that the analytical data used for the TC determination are of known and desired quality, all activities associated with sampling and analysis are conducted under strict Quality Assurance, Quality Control, Chain of Custody procedures. Approved methods for sampling and analysis operations are followed in fulfilment of all regulatory requirements to maintain accuracy, precision, and prevention of bias. This ensured reliability of the data.

Samples are collected either from transport trucks, residue ash conveyor, or from ash pile at intervals of 8 hours, during different operating shifts until a 24-hour composite ash sample was completed. A procedure for random sample grabs under supervision of a knowledgeable shift Operating Engineer is enforced with another person designated as Quality Leader. The composite ash sample is separated into aggregates, unburnts (paper, cardboard, etc.), and unburnables (metals) and weighed separately. Proportionate quantities of the three components are then weighed to make 20 lb. laboratory sample. It is properly labeled, sealed, and stored until sent to a designated and approved laboratory for testing under a Chain of Custody command procedure. An identical 20 lb. sample is prepared to be kept as Archive sample in case the original sample was determined faulty or tempered and had to be rejected. For initial ash characterization, two samples are collected each day for a minimum of one week’s operation of the MSW boilers to yield a total of 14 composite samples.
The standardized sampling procedure used at the Hampton/NASA facility is given in Appendix B.
CHAPTER 3: PRIOR RESEARCH STUDIES

Research studies have indicated use of some of the following ways to achieve the stated objective of controlling leaching of toxic heavy metals from MSW combustion residues.

1. Solidification  
2. Evaporation and vitrification  
3. Stabilization with water-soluble phosphate  
4. Treatment of fly ash with NaOH solutions  
5. Treatment with EDTA solutions  
6. Immobilization with thiourea  
7. Heavy metal stabilization with sodium sulfide

3.1 SOLIDIFICATION

The terms solidification and stabilization can be differentiated by saying that in general while solidification can be called as the conversion of a liquid material into a non-liquid material stabilization generally refers to a chemical reaction introduced for the purpose of making the hazardous constituents in the waste less leachable which are discussed later. Solidification methods reduce the surface area but may or may not necessarily decrease leachability of hazardous substances for which the ash treatment process aims for.

These treatments are among the most widely used processes used for waste incineration residues, mainly the combined APC residue ash (Conner, 1990; Gilliam and Wiles, 1996). The main purpose of solidification/stabilization is to reduce leachability by producing a material with modified physical, mechanical and chemical properties, like specific surface area, durability etc. so that the leachability of contaminants are controlled within the regulatory limits.

Some mechanical separation also plays important role in modifying the physical characteristics of the residue stream. Magnetic and eddy-current separations are used as electromechanical separation processes to reduce its ferrous and non-ferrous metal content primarily from bottom ash. According to the IAWG (1997) and Wiles (1996), the ferrous metal
content of MSWI bottom ash ranges from 7 to 15% by weight, while nonferrous metals account for approximately 1–2% by weight. These would greatly be reduced for Refuse Derived Fuel (RDF) technologies which employ sorting and separation strategies prior to the combustion process. Metal separation from bottom ash may be performed with a view to either metal scrap recovery or to improvement of bottom ash properties for its utilization. Among the chemical separation treatments, simply washing with water is one of the easiest process for removing highly water-soluble constituents from waste incineration residues but it enormously adds up to the volume of waste to be handled and may sometimes not be a preferred method.

Bottom ash is commonly quenched after dropping off the combustion chamber. A high Liquid/Solid ratio and sufficient residence time in the quenching trench may stimulate a reasonably good thermodynamic equilibrium for somewhat effective heavy metal dissolution process. Bottom ash after quenching may still have some residual contents of soluble components. Additional processes of chemical mobilization or aging (IAWG, 1997; Lahl, 1992) may be able to complete the control of heavy metals from leaching beyond desired limits. Salt compounds in the APC residue ash may account for substantial portion of the total ash and are the cause for the negative properties, like high leachability, high water absorption and corrosiveness of such residues. It has been reported that particularly for dry and semi-dry APC residues the high pH of the ash coupled with the large concentrations of highly-soluble heavy metal chlorides are accountable for the partial extraction of such metals as lead, zinc and cadmium during TCLP testing and residue ash needs additional treatment prior to final disposal. Such treatment would include either chemical stabilization or solidification with hydraulic binders.

The most common hydraulic binders include cement, lime and/or pozzolanic materials. However, weak stabilization efficiencies typically have been recorded for soluble salts. Furthermore, due to their strong amphoteric behavior, treatment of zinc and lead with cement- and lime-based processes may be problematic. Chemical stabilization processes have been proposed which basically involve chemical precipitation of heavy metal-incorporating insoluble compounds and/or heavy metal substitution/adsorption into various mineral species. The principal forms of chemical agents used include sulfides (IAWG, 1997; Katsuura et al., 1996), soluble phosphates (Derie, 1996; Eighmy et al., 1997; Hjelmar et al., 1999a, b; Nzihou and
Sharrock, 2002), ferrous iron sulfate (Lundtorp et al., 1999) and carbonates (Hjelmar et al., 1999a, b). Treatments with hydraulic or chemical binders generally yield good leaching properties at relatively low costs.

Leachate composition is the result of reaction between the various mineral phases in the waste and the leaching fluid. The leachability of strongly soluble species (e.g., alkali salts) is almost pH-independent, whereas for a number of contaminants a clear pH-dependence can be observed. The influence of pH on the leaching of contaminants is strongly related to the nature of the particular contaminant under concern as well as the mineral phase(s) in which this is bound. Three main typical leaching behaviors for solubility-controlled leaching have been identified: cation-forming species and non-amphoteric metal ions (e.g. Cd), amphoteric metals (including Al, Pb, Zn), and oxyanion-forming elements (e.g. As, Cr, Mo, V, B, Sb). The concentration of cation-forming species and non-amphoteric metal ions displays fairly constant high values at pH 10.

Figure 13 pH dependency of cation-forming species and non-amphoteric metal ions (Cd)
Figure 14  pH dependency of amphoteric metals (including Al, Pb, Zn)

Figure 15  pH dependency of oxyanion-forming elements (e.g. As, Cr, Mo, V, B, Sb)

Source: Figures 13, 14, 15 Management of municipal solid waste incineration residues,
T. Sabbas, et al.

Other references in these figures are explained below.

Cd (a), Al and Pb (b) and B (c) concentration in eluates and leachate samples of fresh and aged ash (Δ=solidified MSWI residues; O = MSWI bottom ash; □ = MSWI bottom ash + other ashes; X MSWI residues (mixed)) (Sabbas et al., 2001b).
Solidification process comprises of following three principally different techniques that use cement or asphalt as solidification agent.

3.1.1 SOLIDIFICATION OF UNWASHED FLY ASH

Solidification with cement and asphalt are one the traditional methods used for controlling the leachability of Pb and Cd from MSW residue ashes. The major disadvantage of these methods is volume increase of the resulting ash and cement or asphalt mixture besides the added cost of mixing materials used. The resulting mixture has high chlorine and heavy metal contents and therefore a large amount of high quality cement with good hydraulic properties is required. This method has low stabilization efficiency and the resulting residue may deteriorate during long term storage in a landfill. Because of the large amount of solidification agent needed the overall volume of the solidified product increases causing increase in the cost of disposal.
The results in the following table show that mixtures only at high pH levels close to 9 show control of leachability of Pb and Cd in the treated mixture.

Table 8  Effect of pH values on the leachability of heavy metals from the fly ash

<table>
<thead>
<tr>
<th>Metals</th>
<th>pH = 1.5</th>
<th>pH = 3.0</th>
<th>pH = 4.5</th>
<th>pH = 6.0</th>
<th>pH = 7.5</th>
<th>pH = 9.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zn</td>
<td>94.45</td>
<td>88.56</td>
<td>75.43</td>
<td>57.23</td>
<td>28.64</td>
<td>18.65</td>
</tr>
<tr>
<td>Cu</td>
<td>2.2416</td>
<td>1.9567</td>
<td>1.3954</td>
<td>0.71171</td>
<td>0.50362</td>
<td>0.17363</td>
</tr>
<tr>
<td>Pb</td>
<td>45.37</td>
<td>42.36</td>
<td>38.75</td>
<td>24.56</td>
<td>8.327</td>
<td>2.345</td>
</tr>
<tr>
<td>Ni</td>
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<td>0.81150</td>
<td>0.60310</td>
<td>0.3448</td>
<td>0.23151</td>
<td>0.10321</td>
</tr>
<tr>
<td>Cd</td>
<td>2.3459</td>
<td>2.0147</td>
<td>1.7956</td>
<td>1.260</td>
<td>0.84530</td>
<td>0.54281</td>
</tr>
<tr>
<td>Cr</td>
<td>0.22431</td>
<td>0.21242</td>
<td>0.15463</td>
<td>0.13683</td>
<td>0.06254</td>
<td>0.024235</td>
</tr>
</tbody>
</table>

All data in mg/l in the leaching solutions


Besides above observations it is also found that different qualities of cement and asphalt will have different solidification effects as shown by the results in following table.

Table 9  Leachability of the solidified products using different quality of cement and asphalt

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
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<tr>
<td>No. 325 cement</td>
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<tr>
<td>Zn</td>
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<td>0.62590</td>
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<td>0.19435</td>
<td>0.17463</td>
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<td>Pb</td>
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<td>1.0596</td>
<td>1.0286</td>
<td>0.81516</td>
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<td>-----</td>
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<tr>
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<td>0.18290</td>
<td>0.21820</td>
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<table>
<thead>
<tr>
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<th>0.31516</th>
</tr>
</thead>
</table>
| Cd      | 0.014867 | 0.012342 | 0.0086957 | 0.0078541 | 0.0061711 |}

All data in mg/l in the leaching solutions

*Ratio of fly ash to the cement or asphalt (g : g)*


### 3.1.2 SOLIDIFICATION AFTER BASIC WASHING

Using a base chemical for washing transforms soluble heavy metals chlorides into heavy metals hydroxides. These hydroxides precipitate and after filtration and solidification with low quantities of cement they result in a residue with low chlorine contents but with high heavy metals. These heavy metals will be continuously but slowly released to environment.

### 3.1.3 SOLIDIFICATION AFTER ACID WASHING

Washing the residue ash with acid solution results is actually a hydrometallurgical process and it will dissolve most of the heavy metals. No post-solidification treatment with cement or asphalt may be needed.

### 3.2 EVAPORATION AND VITRIFICATION

Removal of heavy metals in fly ash by evaporation at high temperatures has also been practiced. This requires high energy consumption as well as high investments in equipment costs. For these reasons this method is not cost-effective for small and medium size municipal sloid waste incinerators.
3.3 STABILIZATION WITH WATER-SOLUBLE PHOSPHATE

The process uses addition of water soluble phosphate to fly ash and bottom ash residues of municipal solid wastes in order to insolubilize lead and cadmium to an extent as to make the residue in total compliance with EPA regulations. It is claimed that the addition of water-soluble phosphate in residue ashes works for a broad variation in alkalinity of such residues. The water soluble phosphate is either in the form of phosphoric acid, polyphosphoric acid, hypophosphoric acid, metaphosphoric acid or their salts.

The amount of water soluble phosphoric acid to be sprayed is recommended to be about 1 to 8 percent by weight of the acid based on the total ash mixture.

The research is presented in US Patent Number: 4,737,356, date of the patent is April 12, 1988 and it is titled as “Immobilization of lead and cadmium in solid residues from the combustion of refuse using lime and phosphate.” The inventors Mark J. O’Hara and Mario R. Surgi assigned their research to Wheelabrator Environmental Systems.

Some of the results of this experimental study are placed below:

Table 10  Flue Gas Scrubber Product to Fly Ash ratio

<table>
<thead>
<tr>
<th>FGSP: Fly Ash</th>
<th>4:1</th>
<th>4:1</th>
<th>1:1</th>
<th>1:1</th>
<th>3:7</th>
<th>3:7</th>
</tr>
</thead>
<tbody>
<tr>
<td>% H₃PO₄</td>
<td>0</td>
<td>4.25</td>
<td>0</td>
<td>4.25</td>
<td>0</td>
<td>4.25</td>
</tr>
<tr>
<td>EP Toxicity Test</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial pH</td>
<td>12.62</td>
<td>12.24</td>
<td>-</td>
<td>7.40</td>
<td>12.46</td>
<td>5.43</td>
</tr>
<tr>
<td>Final pH</td>
<td>12.38</td>
<td>10.21</td>
<td>5.38</td>
<td>5.05</td>
<td>4.99</td>
<td>5.11</td>
</tr>
<tr>
<td>Extract mg/L</td>
<td>Pb</td>
<td>5.6</td>
<td>0.1</td>
<td>11.8</td>
<td>0.23</td>
<td>8.46</td>
</tr>
<tr>
<td></td>
<td>Cd</td>
<td>0.014</td>
<td>0.01</td>
<td>1.27</td>
<td>0.45</td>
<td>1.33</td>
</tr>
</tbody>
</table>

Source: Mark J. O’Hara M. J., Mario R. Surgi M. R.

Immobilization of lead and cadmium in solid residues from the combustion of refuse using lime and phosphate
The phosphoric acid treatment is shown working well for all 3 tests with 4.25 \% H₃PO₄ treatment with Flue Gas Scrubber Products (FGSP) and fly ash (collected from flue gases in Electrostatic Precipitators or Fabric Filter bags) ratios as 4:1, 1:1, and 3:7.

Table 11  Effect of 4.25\% H₃PO₄ with BA: FA and FGSP: FA ratios

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>FGSP: Fly Ash</td>
<td>4:1</td>
<td>4:1</td>
<td>3:7</td>
<td>3:7</td>
<td>2:1</td>
<td>2:1</td>
<td>1:1</td>
<td>1:1</td>
</tr>
<tr>
<td>% H₃PO₄</td>
<td>-</td>
<td>4.25</td>
<td>-</td>
<td>4.25</td>
<td>-</td>
<td>4.25</td>
<td>-</td>
<td>4.25</td>
</tr>
</tbody>
</table>

EP Toxicity Test

| Initial pH | 12.63 | 12.60 | -   | 7.07 | 12.60 | 12.67 | 12.60 | 12.68 |
| Final pH   | 12.43 | 12.60 | 5.06 | 5.18 | 12.43 | 10.19 | 12.60 | 11.00 |

Extract mg/L

| Pb     | 17.0 | 1.2   | 12.0 | 0.31 | 13.5  | 0.062 | 14.0  | 0.063 |
| Cd     | 0.090| 0.01  | 2.82 | 0.70 | 0.01  | 0.01  | 0.01  | 0.01  |

*Source*: Mark J. O’Hara M. J., Mario R. Surgi M. R., Immobilization of lead and cadmium in solid residues from the combustion of refuse using lime and phosphate

The above table includes effectiveness of 4.25 \% H₃PO₄ treatment of residue ash samples with different BA and FA ratios.

### 3.4 TREATMENT OF FLY ASH WITH NaOH SOLUTIONS

Treating fly ash with sodium hydroxide solutions show that while extraction of lead increases significantly on increasing the pH value or the concentration of NaOH. On the other hand the extraction of Cd either does not change or may increase on increasing the concentration of NaOH as the test results in the following table show.

This chemical is therefore not found suitable for extraction of heavy metals from the fly ash.
Table 12  Leaching of fly ash using NaOH solution \textsuperscript{a, b}

<table>
<thead>
<tr>
<th></th>
<th>N\textsubscript{a}OH concentration (mol/l)</th>
<th>0.1</th>
<th>0.5</th>
<th>1</th>
<th>2</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>\textbf{Pb}</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Concentration in the leaching solution (mg/l)</td>
<td>29.83</td>
<td>36.21</td>
<td>60.98</td>
<td>72.18</td>
<td>85.02</td>
<td></td>
</tr>
<tr>
<td>Pb leached (%)</td>
<td>19.94</td>
<td>24.20</td>
<td>40.76</td>
<td>48.25</td>
<td>56.83</td>
<td></td>
</tr>
<tr>
<td>Content in the leaching residues (mg/kg)</td>
<td>1196</td>
<td>1122</td>
<td>868</td>
<td>763</td>
<td>628</td>
<td></td>
</tr>
<tr>
<td>\textbf{Cd}</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Concentration in the leaching solution (mg/l)</td>
<td>0.53290</td>
<td>0.53316</td>
<td>0.52143</td>
<td>0.50499</td>
<td>0.52917</td>
<td></td>
</tr>
<tr>
<td>Cd leached (%)</td>
<td>20.90</td>
<td>20.91</td>
<td>20.45</td>
<td>19.80</td>
<td>20.75</td>
<td></td>
</tr>
<tr>
<td>Content in the leaching residues (mg/kg)</td>
<td>20.40</td>
<td>20.15</td>
<td>20.27</td>
<td>20.45</td>
<td>20.19</td>
<td></td>
</tr>
</tbody>
</table>

\textsuperscript{a} Weight of the fly ash = 10 g.

\textsuperscript{b} Volume of N\textsubscript{a}OH solution = 100 ml.


Sodium hydroxide dissolves zinc and lead in the ashes and reduces concentration of leachability of these two metals. The possibility of recovery of dissolved metals is one of the advantages of use of this chemical treatment in residue ashes besides this being a very low cost chemical.

The main disadvantage of use of sodium hydroxide is its inability to reduce the leachability concentration of some metals specially cadmium below the regulatory limits.

\section*{3.5 TREATMENT WITH EDTA SOLUTIONS}

A complex agent Ethylenediaminotetraacetate (EDTA) dissolves the soluble salts in the fly ash and is found useful in removing heavy metals from MSW combustion products and thus reduces the leachability of the toxic elements.
The reactions proceed as shown below:

\[
\text{M}^{2+} + \text{N}_2\text{OOCCH}_2\text{N}(-\text{CH}_2\text{CH}_2\text{N})\text{CH}_2\text{COO}\text{N}_a \rightarrow \text{N}_2\text{OOCCH}_2\text{N}(-\text{CH}_2\text{CH}_2\text{N})\text{CH}_2\text{COON}_a
\]

A list of test results using 5 samples given in the table below indicates that over 70% of Pb as well as Cd are leached using EDTA solutions in strengths of 0.1 M or above. The leachability of toxic metals in fly ash can thus be reduced below the regulatory levels.

**Table 13  Effect of treatment of fly ash with EDTA solutions**

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01(^a)</td>
<td>0.02(^a)</td>
<td>0.05(^a)</td>
<td>0.1(^a)</td>
<td>0.2(^a)</td>
<td></td>
</tr>
<tr>
<td><strong>Pb</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Concentration in the leaching solution (mg/l)</td>
<td>27.91</td>
<td>35.74</td>
<td>90.63</td>
<td>108.6</td>
<td>118.2</td>
</tr>
<tr>
<td>Pb leached (%)</td>
<td>18.64</td>
<td>23.89</td>
<td>60.58</td>
<td>72.59</td>
<td>79.01</td>
</tr>
<tr>
<td>Content in the leaching residues (mg/kg)</td>
<td>1226</td>
<td>1137</td>
<td>568</td>
<td>434</td>
<td>314</td>
</tr>
<tr>
<td><strong>Cd</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Concentration in the leaching solution (mg/l)</td>
<td>1.2875</td>
<td>1.3950</td>
<td>1.8020</td>
<td>1.8673</td>
<td>1.9128</td>
</tr>
<tr>
<td>Cd leached (%)</td>
<td>50.49</td>
<td>54.70</td>
<td>70.67</td>
<td>73.23</td>
<td>75.01</td>
</tr>
<tr>
<td>Content in the leaching residues (mg/kg)</td>
<td>12.75</td>
<td>11.47</td>
<td>7.61</td>
<td>6.630</td>
<td>6.375</td>
</tr>
</tbody>
</table>

\(^a\) EDTA (mol/l)

There is seen an appreciable increase in leaching of Pb (60.58 to 72.59%) and Cd (70.6% to 73.23%) if the EDTA concentrations is increased from 0.05 to 0.1 mol/l. This concentration range of EDTA solution is therefore recommended for stabilization and control of Pb and Cd leachability in MSW residue ashes.

The mechanism of EDTA working involves dissolution of most of the heavy metals to below their leachability toxicity without adding much to the volume of treated ashes.

3.6 IMMOBILIZATION WITH THIOUREA

As some sample results show in the table below the leachability of the stabilized metal compounds is below the standard limits even at low concentrations of thiourea, i.e. 0.46 to 0.76% of the fly ash weight, as in samples 1 and 2. The quantities of thiourea needed for stabilization of fly ashes will thus be very low.

Thiourea acts as organic precipitant to form insoluble compounds of heavy metals from the fly ash.

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thiourea added (g)</td>
<td>0.0460</td>
<td>0.0760</td>
<td>0.1649</td>
<td>0.3928</td>
<td>0.7950</td>
<td>1.5345</td>
</tr>
<tr>
<td>Thiourea (mol)</td>
<td>0.00060</td>
<td>0.00100</td>
<td>0.00217</td>
<td>0.00516</td>
<td>0.01044</td>
<td>0.02016</td>
</tr>
<tr>
<td>Thiourea/flyash (wt %)</td>
<td>0.46</td>
<td>0.76</td>
<td>1.65</td>
<td>3.93</td>
<td>7.95</td>
<td>15.34</td>
</tr>
<tr>
<td>C = ([Zn^{2+} + Pb^{2+} + \ldots]) (mol)</td>
<td>1.0301 x 10^{-4}</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Thiourea/C (molar ratio)</td>
<td>5.8</td>
<td>9.7</td>
<td>21</td>
<td>50</td>
<td>101</td>
<td>196</td>
</tr>
<tr>
<td>Concentration in the leaching solution (mg/l)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pb</td>
<td>3.572</td>
<td>1.256</td>
<td>0.9798</td>
<td>0.5589</td>
<td>0.0918</td>
<td>0.08782</td>
</tr>
<tr>
<td>Cd</td>
<td>0.11220</td>
<td>0.10220</td>
<td>0.084152</td>
<td>0.067321</td>
<td>0.039271</td>
<td>0.025245</td>
</tr>
</tbody>
</table>

3.7 HEAVY METAL STABILIZATION WITH SODIUM SULFIDE

The concept of stabilization for heavy metals takes root from the fact that metallic sulfides naturally occur in nature and soluble compounds of heavy metals in combustion ashes can be effectively stabilized by converting them into insoluble sulfides.

The leachability of lead and cadmium of fly ash products stabilized by sulfides is shown in table below. The leachability of Pb and Cd is controlled below the leachability toxicity standards at sodium sulfide concentrations between 0.18% and 0.5% of the fly ash weight and is further reduced at higher dosages of sodium sulfide nonahydrate (Na$_2$S$_9$H$_2$O), or more simply called sodium sulfide hydrate. It is commercially available as Na$_2$S 39%.

Table 15  Stabilization of heavy metals Pb and Cd in MSW fly ash

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na$_2$ S.9H$_2$O added (g)</td>
<td>0.1795</td>
<td>0.5</td>
<td>1</td>
<td>2</td>
<td>4</td>
<td>6</td>
</tr>
<tr>
<td>S$^{2-}$ (mol)</td>
<td>0.00075</td>
<td>0.00208</td>
<td>0.00416</td>
<td>0.00833</td>
<td>0.01665</td>
<td>0.02498</td>
</tr>
<tr>
<td>Sodium sulfide/flyash (wt %)</td>
<td>1.8</td>
<td>5</td>
<td>10</td>
<td>20</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>C = [$Z_{n}^{2+}$ + Pb$^{2+}$ + ….] (mol)</td>
<td>1.0301 x 10$^{-4}$</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S$^{2-}$/C (molar ratio)</td>
<td>7.3</td>
<td>20</td>
<td>40</td>
<td>81</td>
<td>161</td>
<td>243</td>
</tr>
<tr>
<td>Concentration in the leaching solution (mg/l)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pb</td>
<td>7.265</td>
<td>2.737</td>
<td>1.265</td>
<td>0.73712</td>
<td>0.12579</td>
<td>0.10112</td>
</tr>
<tr>
<td>Cd</td>
<td>0.12342</td>
<td>0.10659</td>
<td>0.095372</td>
<td>0.089752</td>
<td>0.053296</td>
<td>0.044881</td>
</tr>
</tbody>
</table>

CHAPTER 4: LEACHABILITY GAP IN HAMPTON RESIDUE ASH

The combustion residue generated at Hampton plant after air pollution control retrofit displayed leachability of toxic substances beyond the regulatory limit and was subjected to remediation and treatment before it could be transported and disposed in landfill.

4.1 LEACHABILITY OF HEAVY METALS IN EXCESS OF REGULATORY THRESHOLD

Leachability of heavy metals especially Cd showed in excess of regulatory threshold when tested as per TCLP method after APC modifications were completed as per EPA emission guidelines.

Some results obtained from TCLP tests during early 2006 after the APC system at Hampton/NASA Steam Plant was upgraded in November 2005 showed Cd and Pb in residue ash were over the regulatory limits. These results are given in Table 7.

Failure to meet the heavy metals leaching and toxicity regulatory limits in residue ashes resulting from the combustion of municipal solid wastes while operating the facility with the modified air pollution control (APC) equipment forced the facility to stop disposing its residue ashes to the designated sanitary landfill located at Big Bethel, Hampton.

The management hired a hazardous material remediation agency to treat and certify that all the accumulated residue ashes at facility’s premises have been converted into non-hazardous and residue ashes no more exhibit any toxicity. These were then disposed of to the landfill after informing State regulators.

The management engaged an agency to design, test and provide a solution to regularly treat the facility’s combustion residue ashes so that the facility could be put back to normal operations after establishing satisfactory treatment procedures. The facility’s Solid Waste permit from the State regulators requires that permittee completes and demonstrates a 14-day testing and characterization of the residue ash to meet the toxicity requirements.
CHAPTER 5: EXPERIMENTAL TESTS AND RESULTS

The results from successive use of different treatment methods as listed below and adopted sequentially at various intervals are discussed in this report:

5.1. Treatment method with a proprietary technology

5.2. Switch over to cost-effective dolomitic lime fines

5.3. Use of dolomitic hydrated lime to replace high calcium hydrated lime for flue gas scrubbing

5.4. Use of increased concentration of high calcium hydrated lime with parametric changes in flue gas scrubbing conditions

5.5. Eliminate use of dolomite taking advantage of alkalinity of boiler process water used for conditioning of fly ash

5.6. Injecting sodium sulfide Na₂S 39% aqueous solution in FA conditioning system

The different treatment methods attempted are discussed in below.

5.1 TREATMENT METHOD WITH PROPRIETARY TECHNOLOGY

The initial trials included 4% concentration by weight of proprietary chemical to the weight of fly ash to be treated while injection rates ranging between 2% and 3% were used during 14-day characterization tests.

The 14-day residue ash characterization results for 7 metals are produced below. Cadmium leached from residue ash at 32.5% of the regulatory threshold of 1mg/L and all other heavy metals were below 1% of their respective threshold limits. The 8th heavy metal mercury was undetectable.
<table>
<thead>
<tr>
<th>METAL</th>
<th>AVERAGE (mg/L)</th>
<th>UCL</th>
<th>Regulatory Threshold (RT)</th>
<th>%RT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>0.03000</td>
<td>0.040</td>
<td>5</td>
<td>0.8%</td>
</tr>
<tr>
<td>Barium</td>
<td>0.08229</td>
<td>0.104</td>
<td>100</td>
<td>0.1%</td>
</tr>
<tr>
<td>Cadmium</td>
<td>0.27769</td>
<td>0.324</td>
<td>1</td>
<td>32.5%</td>
</tr>
<tr>
<td>Chromium</td>
<td>0.00307</td>
<td>0.004</td>
<td>1</td>
<td>0.4%</td>
</tr>
<tr>
<td>Lead</td>
<td>0.02564</td>
<td>0.035</td>
<td>5</td>
<td>0.7%</td>
</tr>
<tr>
<td>Selenium</td>
<td>0.00714</td>
<td>0.00780</td>
<td>1</td>
<td>0.8%</td>
</tr>
<tr>
<td>Silver</td>
<td>0.00129</td>
<td>0.00158</td>
<td>5</td>
<td>0.0%</td>
</tr>
</tbody>
</table>

Source: Hampton Steam Plant data

UCL: Upper Confidence Limit
RT: Regulatory Threshold

For all of the 14 samples tested there were no results that exceeded the applicable regulatory threshold limits.

5.2 SWITCH OVER TO COST-EFFECTIVE DOLOMITIC LIME FINES

The facility conducted research and experimental studies with use of openly available dolomite lime (57.3% Calcium Oxide and 39.7% Magnesium Oxide) in fine particles for treating its combustion fly ash. It initially conducted some in-house tests with use of dolomite fines by 2% to 3% weight ratio of total fly ash to be treated, i.e. 2 tons per day for both boilers operating. After a series of trials were found successfully controlling the leachability of Cd and Pb within the regulatory threshold, the facility continued with conducting a full 14-day continuous testing and characterization of the combined residue ash as required by EPA and the State Solid Waste permit.

The results of the 14-day tests are given in the table below. The cumulative results indicated that during TCLP tests Cd leached at 93.6% of the leachability limit while Pb leached out at 32.2% of the limit.

The Dolomitic Lime Product Information and updated results of heavy metal controls achieved with dolomitic lime treatment are also included in the Appendix B.
Raw data and details of tests carried out by dolomite ash treatment method are given in Appendix D.

Table 17  Residue ash test results with dolomite, Dec. 2008

<table>
<thead>
<tr>
<th>METAL</th>
<th>AVERAGE mg/L</th>
<th>UCL mg/L</th>
<th>RT mg/L</th>
<th>RT %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>0.00271</td>
<td>0.00376</td>
<td>5</td>
<td>0.1%</td>
</tr>
<tr>
<td>Barium</td>
<td>0.30621</td>
<td>0.45668</td>
<td>100</td>
<td>0.5%</td>
</tr>
<tr>
<td>Cadmium</td>
<td>0.76786</td>
<td>0.93618</td>
<td>1</td>
<td>93.6%</td>
</tr>
<tr>
<td>Chromium</td>
<td>0.01086</td>
<td>0.02057</td>
<td>1</td>
<td>2.1%</td>
</tr>
<tr>
<td>Lead</td>
<td>1.06321</td>
<td>1.60970</td>
<td>5</td>
<td>32.2%</td>
</tr>
<tr>
<td>Mercury</td>
<td>0.000507</td>
<td>0.001806</td>
<td>0.2</td>
<td>0.9%</td>
</tr>
<tr>
<td>Selenium</td>
<td>0.00521</td>
<td>0.00756</td>
<td>1</td>
<td>0.8%</td>
</tr>
<tr>
<td>Silver</td>
<td>0.00064</td>
<td>0.00091</td>
<td>5</td>
<td>0.0%</td>
</tr>
</tbody>
</table>

14 Sample Points (includes 4th Quarter Ash Test on 12/16/2008), Hampton Steam Plant data
UCL: Upper Confidence Level
RT: Regulatory Threshold

Some of the TCLP results/data points for Cd control did not fall below the regulatory leachability limits, the cumulative results model was robust, generalizable and defensible even in the face of some outliers lying beyond the averagely drawn trend line. All TCLP test results for Pb had been below its threshold of 5 mg/L.

Routine quarterly testing of residue ash was continued hereafter on a regular basis and cumulative results of all heavy metals were computed based on one-tailed 90% confidence interval as per Student’s T analysis method. With results of the each quarterly test added to compute cumulative values of the leachability controls, the percentage of Cd and Pb leached has continued to decline. The up to date cumulative values of percentage of metals leaching as tested according to TCLP method from a total 28 samples tested since 2009 is given in following table.
Table 18 Summary of results with dolomite use, 12/2015

Table includes residue ash tests results ending 12/2015

<table>
<thead>
<tr>
<th>METAL</th>
<th>AVERAGE mg/L</th>
<th>UCL mg/L</th>
<th>RT mg/L</th>
<th>%RT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>As 0.0114</td>
<td>0.0164</td>
<td>5</td>
<td>0.3%</td>
</tr>
<tr>
<td>Barium</td>
<td>Ba 0.7529</td>
<td>0.8384</td>
<td>100</td>
<td>0.8%</td>
</tr>
<tr>
<td>Cadmium</td>
<td>Cd 0.2716</td>
<td>0.3406</td>
<td>1</td>
<td>34.1%</td>
</tr>
<tr>
<td>Chromium</td>
<td>Cr 0.0404</td>
<td>0.0655</td>
<td>5</td>
<td>1.3%</td>
</tr>
<tr>
<td>Lead</td>
<td>Pb 0.6021</td>
<td>0.9027</td>
<td>5</td>
<td>18.1%</td>
</tr>
<tr>
<td>Mercury</td>
<td>Hg 0.0031</td>
<td>0.0037</td>
<td>0.2</td>
<td>1.9%</td>
</tr>
<tr>
<td>Selenium</td>
<td>Se 0.0054</td>
<td>0.0060</td>
<td>1</td>
<td>0.6%</td>
</tr>
<tr>
<td>Silver</td>
<td>Ag 0.0010</td>
<td>0.0012</td>
<td>5</td>
<td>0.0%</td>
</tr>
</tbody>
</table>

28 Sample points for all 8 metals

Source: Hampton Steam Plant data

5.3 USE OF DOLOMITIC HYDRATED LIME TO REPLACE HIGH CALCIUM HYDRATED LIME FOR FLUE GAS SCRUBBING

At one stage facility had also attempted using hydrated lime with certain percentage of magnesium compound besides calcium oxides for spraying in flue gas scrubber in order to add dolomitic feature in the lime. Two types of dolomitic hydrated limes were considered:

a. Dolomitic Hydrate Type N: Ca(OH)$_2$ 66.7%, MgO 31.8%

b. Dolomitic Hydrate Type S: Ca(OH)$_2$ 61.1%, MgO 37.1%

When using the normal Type N dolomitic quicklime and mixing it with water at atmospheric pressure only the calcium oxide portion of the product will get hydrated as the hydration reaction breaks the quick lime down into fine particles of hydrated lime as per reaction below:
\[
\text{CaO-MgO} + \text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2\text{-MgO}
\]

These products have high neutralizing values and are used for a wide variety of industrial applications like acid neutralization and treatment of hazardous wastes.

In case of dolomitic super hydrate Type S hydration is done at high pressure as magnesium oxide requires high pressure levels or long slaking periods for complete hydration. The reaction takes place as follows:

\[
\text{CaO-MgO} + 2\text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2\text{-Mg(OH)}_2
\]

6 trial tests were carried out by replacing high calcium hydrate for flue gas scrubbing by dolomitic hydrate to fulfill the dual purpose of:

i) Acid scrubbing of flue gases to control \(\text{SO}_2\) emissions (as was otherwise done by use of high calcium hydrate, which was now replaced by dolomitic lime)

ii) Use of magnesium component in the dolomitic lime in scrubber to treat and stabilize resulting fly ash collected downstream of the flue gas treatment process.

Detailed information on these products is provided in Appendix C.

It was noticed that sulfur dioxide emissions were mostly controlled within the required limits though not to the extent as was normally done by use of high calcium (96%) hydrated lime and the leachability of heavy metals (Pb and Cd) in the fly ashes were only partially controlled. Out of the 6 tests conducted 4 did not control the leachability of Pb and Cd to within regulatory threshold. In order to overcome this, the facility attempted to increase the dolomitic lime injection rate in scrubber but the lime slurry injection system was not found supporting extra flows. The experiment was therefore suspended until the facility could upgrade the pumps and re-pipe the slurry discharge to enhance its capacity. Dolomitic Hydrate S (Super hydrate) use was not exercised. Summary results of the 6 tests are given in the table below.
<table>
<thead>
<tr>
<th>METAL</th>
<th>AVERAGE mg/L</th>
<th>UCL mg/L</th>
<th>RT mg/L</th>
<th>%RT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>As</td>
<td>0.01350</td>
<td>0.02157</td>
<td>5</td>
</tr>
<tr>
<td>Barium</td>
<td>Ba</td>
<td>0.48550</td>
<td>0.57541</td>
<td>100</td>
</tr>
<tr>
<td>Cadmium</td>
<td>Cd</td>
<td>0.98587</td>
<td>1.22243</td>
<td>1</td>
</tr>
<tr>
<td>Chromium</td>
<td>Cr</td>
<td>0.06375</td>
<td>0.12123</td>
<td>5</td>
</tr>
<tr>
<td>Lead</td>
<td>Pb</td>
<td>6.25650</td>
<td>9.21424</td>
<td>5</td>
</tr>
<tr>
<td>Mercury</td>
<td>Hg</td>
<td>N/A</td>
<td>N/A</td>
<td>0.2</td>
</tr>
<tr>
<td>Selenium</td>
<td>Se</td>
<td>0.00500</td>
<td>0.005000</td>
<td>1</td>
</tr>
<tr>
<td>Silver</td>
<td>Ag</td>
<td>0.00125</td>
<td>0.00166</td>
<td>5</td>
</tr>
</tbody>
</table>

6 Sample Points for Cd and Pb, 4 Sample Points for other metals (Hg was not tested)

Source: Hampton Steam Plant data

5.4 USE OF INCREASED CONCENTRATION OF HIGH CALCIUM HYDRATED LIME WITH PARAMETRIC CHANGES IN FLUE GAS SCRUBBING CONDITIONS

Another option tried was to inject increased amounts of high calcium (96%) hydrated lime slurry in the flue gas scrubber to find out if added lime that remains unreacted in the scrubber would react with fly ash downstream and thus would be helpful in stabilizing the heavy metals in the combined ash to reduce their leachability as tested with TCLP procedure. Details of results obtained during these tests were mixed and are included in the Appendix.

A brief description of experimental trials carried out for 3 to 4 months during 2013 is given here and a summary of results is provided in the table below.

The amount of lime injected into flue gas scrubber was increased in two ways:
a. Lime flow rates of lime slurry pumps were gradually increased from 56 lb per hour (pph) to 90-95 pph during some tests, while it was kept low at 20 pph in 2 tests
b. The concentration of lime slurry was raised from 1.03 to 1.06/1.08

An operational control change was also made during some of the later tests by gradually raising the Fabric Filter (FF) inlet temperature from 325°F to 400°F.

7 trial tests were conducted with above settings. Results of these tests indicated the following set of results:

1. 2 tests marginally controlled Cd within a tab above the limit, and controlled Pb within limits, while the FF inlet temp was low at 325°F
2. 2 tests controlled Cd at 126% and 128% of limit, both however controlled Pb in limits, again while the FF inlet temp was lower than 400°F
3. 3 later tests were found to effectively control leaching of Cd and Pb within regulatory limits when the FF inlet temp was kept raised to 400°F

Results of 3 other tests conducted with lower concentrations of lime slurry and lower slurry flow rates are not included in these results as they did not control Cd well while Pb was controllable within limit.

It can be summarized from the above results that higher concentrations of high calcium hydrated lime slurry alone may be able to control leachability of both Cd and Pb 50% of the time even at lower FF inlet temperatures and even more effectively at higher FF inlet temperatures of 400°F and above.

It has also helped reduce cooling water requirement in scrubber to a very large extent thus effecting substantial savings in facility’s water bill.
In place of current use of high calcium hydrated lime slurry in the countercurrent spray tower, a newer product the magnesium-enhanced lime process (MEL) can be more effectively as it is a variation of the lime process in that it uses a special type of lime: magnesium-enhanced lime (typically 5% – 8% magnesium oxide) or dolomitic lime (typically 20% magnesium oxide). The MEL process may be designed to utilize the alkalinity of fly ash in addition to the alkalinity of a sorbent. Lime used in the MEL contains magnesium in addition to its calcium component. Because of the greater solubility of magnesium salts compared to calcium sorbents, the scrubbing liquor is significantly more alkaline. Therefore, MEL is able to achieve high SO₂ removal efficiencies in significantly smaller absorber towers than the limestone.
scrubbers. Additionally, MEL allows for a significant decrease of liquid/gas (L/G) ratio, compared to high calcium hydrated lime for a given SO$_2$ removal target. This chemical has not been tried at the study site but is recommended as an alternative to the in-line dolomitic lime injection downstream.

5.5 ELIMINATE USE OF DOLOMITE TAKING ADVANTAGE OF ALKALINITY OF BOILER PROCESS WATER USED FOR CONDITIONING OF FLY ASH

Over the years the facility has changed the source of water used for conditioning the fly ash in the screw conveyor that moves the fly ash onto the vibrating pan at a point where the fly ash mixes with the bottom ash being carried form the furnace bottom. The initial source of water mixed for fly ash conditioning in the conveying screw was the city water at ambient temperature. The water was able to condition the fly ash to avoid it being air-borne, but it converted the ash into cement like slurry and ultimately had very detrimental effect on the life of the conditioning screw. Because of frequent failures of conditioning screw in trying to move cementitious ash, the facility has started recycling and utilizing conditioning water from the boiler process blowdown system which is at higher temperature and is no longer resulting in cementing of the fly ash, besides effecting huge savings in water consumption.

The innovative use of hot boiler bow down process water for fly ash conditioning is also providing a source of additional alkalinity to the residue ash and it can be safely assumed that it is helping in maintaining a better pH balance in the residue ashes which in turn is leading to better stabilization of heavy metals.

A set of 5 tests were performed giving consideration to the above aspect of mixing of boiler process water in fly ash for its conditioning. During these tests the system operational variant of setting up the temperature at which the flue gases exit the SDA and then are passed on to the fabric filters for fly ash collection was further changed up from 400$^\circ$ F to 430$^\circ$ F. The lime slurry flows to the scrubber were however kept as normal and low, and reagent specific gravity was also lowered to 1.03.
The results of the 5 tests carried out with above settings are tabulated below.

The concept of utility of boiler blow down process water in fly ash conditioning coupled with changes in flue gas scrubber operational settings did not seem to be controlling the leachability of either Cd or Pb in any uniform way.

Table 21 Summary results of treatment by high calcium hydrated lime, 11/2015

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Sample Date</th>
<th>As</th>
<th>Ba</th>
<th>Cd &lt; 1 mg/L</th>
<th>Cr</th>
<th>Pb &lt; 5 mg/L</th>
<th>Hg</th>
<th>Se</th>
<th>Ag</th>
</tr>
</thead>
<tbody>
<tr>
<td>HSP-815-C1</td>
<td>8/19/15</td>
<td>1.260</td>
<td></td>
<td>9.012</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HSP-915-C2</td>
<td>9/12/15</td>
<td>1.450</td>
<td></td>
<td>3.940</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HSP-915-C3</td>
<td>9/12/15</td>
<td>0.969</td>
<td></td>
<td>8.050</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HSP-915-C4</td>
<td>10/29/15</td>
<td>1.490</td>
<td></td>
<td>25.500</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HSP-915-C5</td>
<td>11/12/15</td>
<td>0.005</td>
<td>0.483</td>
<td>1.020</td>
<td>0.001</td>
<td>0.0127</td>
<td>0.000</td>
<td>0.009</td>
<td>0.001</td>
</tr>
</tbody>
</table>

These tests were conducted at low normal lime slurry flows and low reagent specific gravity.

Source: Hampton Steam Plant data

5.6 INJECTING SODIUM SULFIDE Na₂S 39% AQUEOUS SOLUTION IN FLY ASH CONDITIONING SYSTEM

The practice of using dolomitic fines had been continued while the facility carried out its attempts to find other options as well. The base price of dolomitic lime is affordable, but the current packing and transportation costs in 2 ton super sacks costs the facility about the same as the cost of chemical itself. The management of the facility weighed-in following options to overcome it:

a. Construct a storage silo large enough to store long-term supplies of dolomite lime transported in bulk trucks to avoid paying heavily for supply in super sacks.

b. Make process/chemical use changes upstream of fly ash generation, for example to increase spraying of high calcium hydrated lime (which is stored in a silo and mixed with water to make slurry) or to spray a mix of high calcium hydrated lime and dolomite lime in the flue gases to ascertain if it will change the reaction kinetics to the extent that may help eliminate use of dolomite lime in the fly ash collection system downstream of the flue gas path.
c. Find an alternative to dolomite lime in form of a liquid chemical injection that would use an existing process water injection as part of fly ash conditioning. A small liquid storage tank and pump would be needed for this system in case it is determined to treat the ashes.

Initial trials with sodium sulfide liquid (Na$_2$S 39%) chemical injection as part of proposal in item c. above was undertaken. Results and validity of the results of trials were evaluated to find out if they meet the stabilization criteria of heavy metals.

The results of leachability of Cd and Pb of a test carried out on Feb 3, 2016 are given in the table below.

Table 22  Leachability of Cd and Pb, Sodium sulfide test on Feb 3, 2016

Source: Laboratory results for Hampton Steam Plant residue Sample ID: HSP-0216-SS1

Total 5 such trial tests were conducted during Feb 3 and March 3, 2016. The results indicated that the leachability of both Cd and Pb has been found below the regulatory limits. The MSW residue ash was treated with an estimated injection rate of 15 gallons per day. Results of these tests are further analyzed in Chapter 8.
CHAPTER 6: DISCUSSIONS AND ANALYSIS OF RESULTS OBTAINED DURING USE OF PROPERIETARY COMPOUND AND DOLOMITE

The study used the three principles of experimental design at all stages, starting from ensuring the replication of sample data by resorting to sound engineering controls. Thus it avoided the errors, biases and noises in sampling data. All necessary quality controls were exercised in gathering, preparing, securing, and transporting the samples following well-written and strictly followed procedures and under an established chain of custody command. The sample data were completely randomized by assigning treatments and factoring for all applicable running conditions of the combustion units so that the results of the sample can be elevated to the system level.

With several set of experimental data available for analysis a matrix of causal effects of various Treatment methods is created adding different running conditions of the 2 boiler units as blocks to generate a Randomized Complete Block (RCB) design.

A statistical analysis carried out with the results of first 4 treatment methods that are broadened up to 6 treatment options T1 through T6 and 4 running conditions RC1 through RC4 were used as blocks. The last 2 treatment methods have not been included this analysis as these were either not concluded due to under capacity of the reagent slurry pumping system to the flue gas scrubber or a 6th treatment method number 13.6 was still under way while writing this report and did not have substantial number of test results to be included.

A total of 102 results tested for Cd and Pb at an approved laboratory have been included in this statistical analysis. Mass flux changes during boilers running conditions variability is used in this analysis. Shapiro-Wilk tests for normality at a higher 95% confidence level and comparisons of scatter graphs did not reveal normality of data as probability values of both Cd and Pb were < 0.001. This would be mainly due to the fact that some of the treatment methods are observed controlling the target limit very differently between various tests and trials.
Table 23  ANOVA: Description of Treatment methods and Running Conditions

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>Proprietary 1</td>
</tr>
<tr>
<td>T2</td>
<td>Proprietary 2</td>
</tr>
<tr>
<td>T3</td>
<td>Proprietary 3</td>
</tr>
<tr>
<td>T4</td>
<td>Proprietary 4</td>
</tr>
<tr>
<td>T5</td>
<td>Dol. Lime 2.5%</td>
</tr>
<tr>
<td>T6</td>
<td>Dol. Lime 2%</td>
</tr>
</tbody>
</table>

Notes:

- Test Duration: Test duration is homogeneous (24 hours) for all the treatments and Running Conditions (RC)
- RC1: Both boilers in continuous running condition
- RC2: One boiler in continuous running condition
- RC3: 1 boiler is running continuously for 24 hours + 1 boiler is starting up
- RC4: 1 boiler is running continuously for 24 hours + 1 boiler is shutting down
- Conc: Concentration is listed in mg/L

Table 24  ANOVA data entries

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Description</th>
<th>RC1</th>
<th>RC2</th>
<th>RC3</th>
<th>RC4</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>Proprietary 1</td>
<td>12, 73</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T2</td>
<td>Proprietary 2</td>
<td>13, 22, 59</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T3</td>
<td>Proprietary 3</td>
<td>396, 481, 296, 659, 47, 736, 422, 638, 896, 775</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T4</td>
<td>Proprietary 4</td>
<td></td>
<td>422, 736</td>
<td></td>
<td>896, 676, 805</td>
</tr>
<tr>
<td>T5</td>
<td>Dol. Lime 2.5%</td>
<td></td>
<td></td>
<td>178, 315, 35</td>
<td>347, 272, 164, 306</td>
</tr>
</tbody>
</table>

Notes:

- Test Duration: Test duration is homogeneous (24 hours) for all the treatments and Running Conditions (RC)
- RC1: Both boilers are used in continuous running condition
- RC2: One boiler used in continuous running condition
- RC3: 1 boiler is running continuously for 24 hours + 1 boiler is starting up
- RC4: 1 boiler is running continuously for 24 hours + 1 boiler is shutting down

Source: Table of 102 data sets for Treatments and Running Conditions for ANOVA analysis
As a choice of factors, six treatment methods (T1, T2, T3, T4, T5, T6) under four different boiler running conditions (RC1, RC2, RC3, RC4) are used concurrently for control of concentrations of both the above two heavy metals and results are tested according to the following Test of Hypotheses for each of these two heavy metals.

Control of Cd leachability < 1.0 mg/L and control of Pb leachability < 5.0 mg/L (both tested concurrently under TCLP procedure and with EPA Method 1311) using 6 treatments and 4 running conditions of the two boilers in use at Hampton/NASA Plant.

All samples were collected over an extended number of hours as per an approved and established procedure from the residue ash generated over the previous 24-hour period.

The total number of samples used in the study was spread over several years while different treatment methods were either experimented, or were being proven, or were otherwise used for regular mandated quarterly testing of residue ash.

Following is the total number of samples used for the two constituents:

A. Cadmium 102 samples
B. Lead 102 samples

Samples collected were used for analyzing the concentrations of both toxic pollutants simultaneously.

As a plan for selection of responsible variable, Treatments T1 through T6 were chosen as primary independent variable, while running conditions of the set of two boilers were considered as secondary independent variable affecting the outcomes.

Following choices for experimental designs were used.

(A) Cadmium
F-test based T.H. at 95 % confidence (α = 0.05):

Hypothesis #1: (Test of treatment effects: T1 through T6)
H₀: τ₁ = τ₂ = τ₃ = τ₄ = τ₅ = τ₆ = 1 ppm
H₁: At least one of τ₁ through τ₆ < 1 ppm

Hypothesis #2: (Test of block effects: RC1 through RC4)
H₀: β₁ = β₂ = β₃ = β₄ = 1 ppm
H₀: At least one of β₁ through β₄ < 1 ppm

(B) Lead
F-test based T.H. at 95 % confidence (α = 0.05):

Hypothesis #1: (Test of treatment effects: T1 through T6)
H₀: τ₁ = τ₂ = τ₃ = τ₄ = τ₅ = τ₆= 5 ppm
Hₐ: At least one of τ₁ through τ₆ < 5 ppm

Hypothesis #2: (Test of block effects: B1 through B4)
H₀: β₁ = β₂ = β₃ = β₄ = 5 ppm
Hₐ: At least one of β₁ through β₄ < 5 ppm

The source of sample data was the fly ash generated at the Waste-to-Energy Hampton/NASA Steam Plant from the combustion process of one or both the operating boilers. It was treated with varying concentrations of different chemicals and representative ash samples were prepared for analysis of heavy metal constituents. The samples were randomly grabbed either from the ash pile, one half of front loader bucket from each truck being loaded for ash disposal, or directly from the ash dumping conveyor at set hourly intervals if the trucks were being loaded directly with ash for disposal to landfills. The total ash grab was then quartered, a single quarter was selected by random coin toss, and that would then be separated into three components: aggregate, paper/cardboard etc. (unburnts), and metals (unburnables). Each component was weighed separately and proportionate weight of each of the three components was calculated and weighed to make a composite 20 lb. sample, all under expert supervision or by a trained quality leader. Two such 20 lb. samples were prepared, one to be tested and the other kept as archive sample in case the original sample got damaged/pilfered or judged unusable, both 20 lb. samples were sealed with forensic tape, signed, and authenticated by the quality leader.

The samples were prepared as described above, kept under control of responsible official, and were then sent to an approved laboratory (or, alternatively were collected by the lab’s
representative) all under an approved and established chain of custody procedure to ensure safety and security of the collected samples. The sample preparation methodology and analysis is based on EPA guidelines laid out in Solid Wastes Procedure and method SWP-846.

Regression modelling and validation of data distribution was carried out by drawing the curves.

The respective regression curves of the two target pollutants Cd and Pb resembled following shapes:

A. Cd  Power model
B. Pb  Exponential model

The concentration data of these two pollutants were transferred to power and exponential terms, respectively and the resulting regression curves are shown below.

![Cd Trendline](image)

**Figure 16**   Power and exponential curves for Cd for 102 samples studied

*Source:* 102 data points for Cd from Hampton test results
Re-runs showed no normality of the data distribution for any of the two pollutants. Besides above models, histograms for both Cd and Pb were drawn as below.

Source: 102 data points for Pb from Hampton Steam Plant test results

Source: 102 data points for Cd from Hampton Steam Plant test results
Since the number of samples for both Cd and Pb were fairly large, 102 in each case, the sampling distribution of the sample mean is considered approximately normally distributed according to Central Limit Theorem (CLT). This was further verified using Analysis of Variance (ANOVA) procedure.

No data have been considered for filtering in this analysis. There are some concentrations above normal and high outcomes are judged as part of the exploratory testing to find out appropriate treatment for controlling the concentrations of pollutants leached out. These results have therefore been also included in the analysis of variance (ANOVA) study.

The results of the Sample Data, SAS Estimates and ANOVA Output are included in Appendix D of this study.

Following assumptions were made while conducting this study:

1. The municipal solid waste (MSW) used for combustion process is considered mostly of uniform characteristics throughout the year. It is delivered to the boilers in as-received condition.
2. The two combustion units (boilers) are identical and operate with similar mass flux rates of fuel inputs and residue ash output.

3. The treatment chemicals used during their respective test duration were qualitatively and qualitatively uniform during the entire period.

4. The samples came from a normally distributed system.

The experimental design in the study uses more than two treatments that are the factor of interest has more than two levels (in fact the study is using 6 treatments). The blocks used are significant variables in the sense that while only one boiler is running in steady state, the total mass flux of the combined residues sharply varies quantitatively as well as qualitatively with variations occurring every time the second boiler is shutting down with sharply lowering gradient in flue gas temperature and quantities. Opposite to this, if one boiler is running in steady state but the second boiler is starting up, the total mass flux of the combined residue is experiencing a sharp up-gradient for several hours in flue gas temperatures and quantities.

The study follows the procedure that meets the standards for randomized complete block design by running a complete replicate of the treatment in each block because the actual assignments of each of the 6 treatments are done randomly in each block.

The SAS System ANOVA Procedure table showed the following F- and p-values for the combined ash (CA) treatments and blocks for Cd and Pb, respectively:

<table>
<thead>
<tr>
<th>Analysis/Result</th>
<th>F-value</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reject null hypothesis</td>
<td>12.69</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Fail to reject null hypothesis</td>
<td>2.11</td>
<td>0.1040</td>
</tr>
</tbody>
</table>

For control of Cd concentrations at 95% level of confidence, the p-value for chemical treatments < 0.05, there is significant evidence that one or more of the treatments are immobilizing and controlling the leachability concentration of Cd. But since the p-value for boilers running conditions is > 0.05, the study fails to reject the null hypothesis and therefore
concludes that the boilers running conditions do not have any significant influence on the stabilization and control of Cd leachability concentration.

<table>
<thead>
<tr>
<th></th>
<th>F-value</th>
<th>p-value</th>
<th>Analysis/Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>B. Pb</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CA Treatment</td>
<td>14.01</td>
<td>&lt;0.001</td>
<td>Reject null hypothesis</td>
</tr>
<tr>
<td>CA Bulk</td>
<td>0.93</td>
<td>0.4292</td>
<td>Fail to reject null hypothesis</td>
</tr>
</tbody>
</table>

For control of Pb concentrations at 95% level of confidence, the p-value for chemical treatments < 0.05, there is significant evidence that one or more of the treatments are immobilizing and controlling the leachability concentration of Pb. However, since the p-value for boilers running conditions is > 0.05, the study fails to reject the null hypothesis and therefore concludes that the boilers running conditions do not have any significant influence on the stabilization and control of Pb leachability concentration.

Detailed discussions on the next study of use of aqueous sodium sulfide for treatment of Hampton facility’s residue ash are included in Chapter 8.
CHAPTER 7: COAL COMBUSTION RESIDUES

U.S. coal-fired power plants generate approximately 100 million tons of coal ash annually, and 75% of this is in form of fly ash. Coal combustion residues (CCRs) result from the combustion of coal in steam generating and electric power plants. The residues include coal fly ash and bottom ash and also waste from flue gas desulfurization (FGD) in the electricity generating units (EGUs). Steam electric power plants use variety of fuels including nuclear and fossil fuels such as coal, oil, and natural gas and discharge large quantities of wastewaters. They carry both toxic and bioaccumulative pollutants including arsenic, mercury, selenium, chromium, and cadmium accounting for about 30% of all toxic pollutants that are discharged into surface waters and are governed by Clean Water Act (CWA). This study includes wastewater discharges from coal power plants only.

Recently new processes like coal gasification and clean coal technologies have been introduced for generating electric power from coal and new pollution control measures, like new technologies for flue gas desulfurization (FGD) and flue gas mercury control (FGMC) have been implemented. These have changed the nature of coal power plant waste streams. As a result the toxic pollutants in the coal power plant wastewater discharges are a concern for public health and environment. Toxic metals like mercury, arsenic, lead, and selenium accumulate in fish and contaminate drinking water. The effects of these pollutants can cause cancer, cardiovascular diseases, neurological disorders, and kidney and liver damage.

7.1 REGULATIONS GOVERNING COAL COMBUSTION RESIDUES

Regulations requiring safe disposal of coal combustion residues (CCRs) are relatively recent. EPA finalized the national regulations for safe disposal of CCRs from coal power plants on April 17, 2015. The rules include the technical requirements for CCR landfills and surface impoundments under Resource Conservation and Recovery Act (RCRA) Subtitle D that regulates solid wastes. The rules were the results of extensive study on the effects of coal ash on the environment and public. During the study, EPA also found that the use of wet FGD systems to control sulfur dioxide (SO₂) emissions has increased significantly since the last revision of the effluent guidelines in 1982. It was also estimated that its use will continue to increase after the
steam electric power generating units are taking steps to address federal and state air pollution control requirements. FGD wastewaters were generally found to contain significant levels of metals and other pollutants. While advanced treatment technologies are available to treat the FGD wastewater, however most plants were still using surface impoundments designed primarily to remove suspended solids from FGD wastewater. It has also been determined that technologies are available for handling the fly ash and bottom ash generated at a plant without using any water or at least eliminating the discharge of any ash transport water. The waters used to convert fly ash and bottom ash into slurry form to transport these wastes are generated in large quantities from wet systems at coal-fired power plants and contain significant concentrations of metals, including arsenic and mercury.

The Disposal of Coal Combustion Residuals from Electric Utilities final rule (the “Coal Ash Rule,” or the “Rule”), signed December 19, 2014, sets first-ever minimum federal standards for the disposal of coal ash under the Resource Conservation and Recovery Act (RCRA). Through this rulemaking, EPA has elected to classify coal ash as a non-hazardous solid waste subject to regulation under subtitle D of RCRA. This means that the federal government cannot enforce the rule, and cannot mandate that states adopt and enforce the federal standards. EPA “strongly encourages states to revise their Solid Waste Management Plans to implement the standards.” Because the Rule is not enforceable by EPA, and state enforcement is uncertain, a primary enforcement mechanism for the Rule is citizen suits under RCRA. Other standards, such as those found in the Clean Water Act, still apply to coal ash.

One of the major provisions of the Rule is that it calls for the closure of surface impoundments and landfills that fail to meet engineering and structural standards, and regular inspections of the structural safety of surface impoundments. New surface impoundments and landfills will also be restricted to locations not deemed “sensitive,” such as wetlands and earthquake zones. The rules also call for use of fugitive dust controls to reduce windblown coal ash dust, and liner barriers for new units and proper closure of surface impoundments and landfills that will no longer receive CCRs. The final rule means that states must now revise their Solid Waste Management Plans (SWMPs) and submit these revisions to the EPA for approval. “A revised and approved SWMP will signal EPA’s opinion that the state SWMP meets the federal criteria,” the EPA said.
The rule applies to all active landfills and ponds, but it does not apply to following:

a. The placement of coal ash in coal mines

b. Coal ash landfills that ceased receiving coal ash prior to the effective date of the Rule

c. Coal ash units at facilities that have ceased producing electricity prior to the effective date of the Rule

d. Practices that meet the definition of “beneficial use” of coal ash (< 12,400 tons of fill) or any type of past beneficial uses

e. The disposal of coal ash from non-utility boilers burning coal (e.g., paper plants, industrial boilers generating electricity for their own use, university power plants, etc.)

The current rule covering disposal of coal combustion residues has a number of deficiencies:

1. Treats coal ash as a nonhazardous solid waste rather than a hazardous waste, thus regulating coal ash under subtitle D rather than subtitle C

2. Relies on states voluntarily adopting standards and citizen suits for enforceability

3. Continues to allow coal ash to be stored in unlined ponds. Unlike the proposed rule, the final Rule does not call for the lining or closure of all coal ash ponds within 5 years

4. Only requires assessment work to be done by a “qualified professional engineer,” not an independent engineer

5. There are no groundwater protection standards for: aluminum, boron, chloride, copper, iron, manganese, pH, sulfate, sulfide, and TDS, so high levels of these pollutants will not trigger corrective action

6. All inactive landfills are not regulated

7. Inactive ponds at inactive power plants are not regulated

8. Closure deadlines provide for multi-year extensions

9. Inactive coal ash ponds closed in the next 3 years will require no post-closure care requirements such as groundwater monitoring and corrective action
10. No specific standards for particulates in the air at coal ash plants

11. Structural fill that is less than 12,400 tons does not require an affirmative demonstration in order to be considered beneficial use.

### 7.2 PHYSICAL CHARACTERISTICS OF COAL ASH

Coal combustion residues have been studied in detail on the following aspects:

- The process of formation of coal ash
- Coal ash characteristics
- The way the coal ash weathers in the environment

Typically coal ash also has the same components like the various steams generated from combustion of MSW – fly ash from ESP or Fabric filters, flue gas desulfurization (FGD) ash form flue gas cleaning, and bottom ash or boiler slag.

Fly ash from coal combustion is formed when molten minerals such as clay, quartz, and feldspar, solidify in the moving air stream, giving approximately 60% of the fly ash particles a spherical shape. Coal fly ash is a pozzolanic material (as used for concrete production) and has been classified into two classes, F and C, based on the chemical composition of the fly ash.

According to ASTM C 618, the chemical requirements to classify any fly ash are shown in the following table.

<table>
<thead>
<tr>
<th>Properties of fly Ash Class</th>
<th>Class F</th>
<th>Class C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Silicon dioxide (SiO₂) plus aluminum oxide (Al₂O₃) plus iron oxide (Fe₂O₃), min, %</td>
<td>70.0</td>
<td>50.0</td>
</tr>
<tr>
<td>2. Sulfur trioxide (SO₃), max, %</td>
<td>5.0</td>
<td>5.0</td>
</tr>
<tr>
<td>3. Moisture Content, max, %</td>
<td>3.0</td>
<td>3.0</td>
</tr>
<tr>
<td>4. Loss on ignition, max, %</td>
<td>6.0</td>
<td>6.0</td>
</tr>
</tbody>
</table>
Class F fly ash is produced from burning anthracite and bituminous coals. This fly ash has siliceous or siliceous and aluminous material, which itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperature to form cementitious compounds.

Class C fly ash is produced normally from lignite and sub-bituminous coals and usually contains significant amount of Calcium Hydroxide (CaOH) or lime. This class of fly ash, in addition to having pozzolanic properties, also has some cementitious properties (ASTM C 618-99). Color is one of the important physical properties of fly ash in terms of estimating the lime content qualitatively. It is suggested that lighter color indicate the presence of high calcium oxide and darker colors suggest high organic content.

The primary factors that influence the mineralogy of a coal fly ash are:

1. Chemical composition of the coal
2. Coal combustion process including coal pulverization, combustion, flue gas clean up, and fly ash collection operations
3. Additives used, including oil additives for flame stabilization and corrosion control additives.

The minerals present in the coal dictates the elemental composition of the fly ash. But the mineralogy and crystallinity of the ash is dictated by the boiler design and operation. The pozzolanic reactions are as follows:

\[
\text{Ca(OH)}_2 \rightarrow \text{Ca}^{++} + 2[\text{OH}]^-
\]

\[
\text{Ca}^{++} + 2[\text{OH}]^- + \text{SiO}_2 \rightarrow \text{C-S-H}
\]

(Silica) (Gel)

\[
\text{Ca}^{++} + 2[\text{OH}]^- + \text{Al}_2\text{O}_3 \rightarrow \text{C-A-H}
\]

(Alumina) (Gel)

Hydration of tri-calcium aluminate in the ash provides one of the primary cementitious products in many ashes.

Fly ash particles also contain crystalline compounds that pass through the combustion zone or are formed at high temperatures. Some elements that become volatile at high temperatures, like arsenic and selenium, later condense at the surface of the fly ash particles as
the ash cools. The particles are spherical in shape and are either solid or are with vesicles, as shown in the following figure.

The 6 different characteristics of fly ash particles shown in the figure are described below.

(a) Typical spherical morphology of glassy particles.

(b) A large hollow sphere formed when entrapped gas expanded during thermal decomposition of calcium carbonate (CaCO₃).

(c) A particle etched with hydrofluoric acid to remove surface glass and reveal a shell of interlocking mullite crystals.

(d) A typical magnetic spinel mineral (magnetite) separated from ash after removal of encapsulating glass.

(e) A fractured ash particle containing numerous vesicles.

(f) The accumulation of tiny granules of inorganic oxides, crystals, and coalesced ash on the surface of a larger particle.

In majority of the coal power plants, approximately 80% of the units, removal of sulfur form flue gases are based on lime or limestone wet scrubbing. The remaining utilize either sodium-based or lime slurry (spray) dry scrubbing or use various sorbent injection technologies of one form or another. In the United States, coal-fired utility boilers have been adopting newer and best available control technologies for emission control since they are a major source of SO₂ emissions. In the wet scrubbers the alkaline sorbent reacts with the SO₂ gas and is collected in a
liquid form as calcium sulfite or calcium sulfate slurry. The calcium sulfite or sulfate is allowed to settle out as most of the water is recycled. Stabilized calcium sulfite FGD scrubber material has been used as an embankment and road base material.

The volatile elements (e.g., As, B, Cl, F, S, Se) are found concentrated in the fly ash or FGD sludge.

Bottom ash that falls to the bottom of the furnace is made up of heavier particles and is mainly composed of amorphous and glassy aluminous silicate from the melted mineral phases in coal. Boiler slag is collected in plants that operate at very high temperatures and where the molten particles are cooled and quenched in water. Coal fly ash and bottom ash show similarity in composition and variability of the nonvolatile inorganic elements (e.g., Al, Ca, Fe, and Si). Total concentrations of several elements (e.g., As, B, Pb, Zn) vary with the coal type used in the burning process. Bottom ash accounts for 25% of all coal combustion residues in USA.

### 7.3 CHEMICAL COMPOSITION OF COAL ASH

About 90% of mineral components of coal fly ash are the oxides of silicon, aluminum, iron, and calcium, minor constituents such as magnesium, potassium, sodium, titanium, and sulfur account for about 8% while trace constituents such as arsenic, cadmium, lead, mercury, and selenium, together make up less than 1% of the total composition. Typical range of major and trace constituent concentrations in fly ash, bottom ash, rock, and soil for comparison are shown in Table below.

<table>
<thead>
<tr>
<th>Component</th>
<th>Fly Ash</th>
<th>Bottom Ash</th>
<th>Rock</th>
<th>Soil</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>70,000 – 140,000</td>
<td>59,000 – 130,000</td>
<td>9,800 – 96,000</td>
<td>15,000 – 100,000</td>
</tr>
<tr>
<td>Calcium</td>
<td>7,400 – 150,000</td>
<td>5,700 – 150,000</td>
<td>6,000 – 83,000</td>
<td>1,500 – 62,000</td>
</tr>
<tr>
<td>Iron</td>
<td>34,000 – 130,000</td>
<td>40,000 – 160,000</td>
<td>8,800 – 95,000</td>
<td>7,000 – 50,000</td>
</tr>
<tr>
<td>Silicon</td>
<td>160,000–270,000</td>
<td>160,000–280,000</td>
<td>57,000–380,000</td>
<td>230,000–390,000</td>
</tr>
<tr>
<td>Magnesium</td>
<td>3,900 – 23,000</td>
<td>3,400 – 17,000</td>
<td>700 – 56,000</td>
<td>1,000 – 15,000</td>
</tr>
<tr>
<td>Potassium</td>
<td>6,200 – 21,000</td>
<td>4,600 – 18,000</td>
<td>4,000 – 45,000</td>
<td>4,500 – 25,000</td>
</tr>
<tr>
<td>Element</td>
<td>Range</td>
<td>Median Range</td>
<td>Lower Range</td>
<td>Upper Range</td>
</tr>
<tr>
<td>----------</td>
<td>-------------</td>
<td>--------------</td>
<td>-------------</td>
<td>-------------</td>
</tr>
<tr>
<td>Sodium</td>
<td>1,700 – 17,000</td>
<td>1,600 – 11,000</td>
<td>900 – 34,000</td>
<td>1,000 – 20,000</td>
</tr>
<tr>
<td>Sulfur</td>
<td>1,900 – 34,000</td>
<td>BDL – 15,000</td>
<td>200 – 42,000</td>
<td>840 – 1,500</td>
</tr>
<tr>
<td>Titanium</td>
<td>4,300 – 9,000</td>
<td>4,100 – 7,200</td>
<td>200 – 5,400</td>
<td>1,000 – 5,000</td>
</tr>
<tr>
<td>Antimony</td>
<td>BDL – 16</td>
<td>All BDL</td>
<td>0.08 – 1.8</td>
<td>BDL – 1.3</td>
</tr>
<tr>
<td>Arsenic</td>
<td>22 – 260</td>
<td>2.6 – 21</td>
<td>0.50 – 14</td>
<td>2.0 – 12</td>
</tr>
<tr>
<td>Barium</td>
<td>380 – 5100</td>
<td>380 – 3600</td>
<td>67 – 1,400</td>
<td>200 – 1,000</td>
</tr>
<tr>
<td>Beryllium</td>
<td>2.2 – 26</td>
<td>0.21 – 14</td>
<td>0.10 – 4.4</td>
<td>BDL – 2.0</td>
</tr>
<tr>
<td>Boron</td>
<td>120 – 1000</td>
<td>BDL – 335</td>
<td>0.2 – 220</td>
<td>BDL – 70</td>
</tr>
<tr>
<td>Cadmium</td>
<td>BDL – 3.7</td>
<td>All BDL</td>
<td>0.5 – 3.6</td>
<td>BDL – 0.5</td>
</tr>
<tr>
<td>Chromium</td>
<td>27 – 300</td>
<td>51 – 1100</td>
<td>1.9 – 310</td>
<td>15 – 100</td>
</tr>
<tr>
<td>Copper</td>
<td>62 – 220</td>
<td>39 – 120</td>
<td>10 – 120</td>
<td>5.0 – 50</td>
</tr>
<tr>
<td>Lead</td>
<td>21 – 230</td>
<td>8.1 – 53</td>
<td>3.8 – 44</td>
<td>BDL – 30</td>
</tr>
<tr>
<td>Manganese</td>
<td>91 – 700</td>
<td>85 – 890</td>
<td>175 – 1400</td>
<td>100 – 1,000</td>
</tr>
<tr>
<td>Mercury</td>
<td>0.01 – 0.51</td>
<td>BDL – 0.07</td>
<td>0.1 – 2.0</td>
<td>0.02 – 0.19</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>9.0 – 60</td>
<td>3.8 – 27</td>
<td>1.0 – 16</td>
<td>All BDL</td>
</tr>
<tr>
<td>Nickel</td>
<td>47 – 230</td>
<td>39 – 440</td>
<td>2.0 – 220</td>
<td>5 – 30</td>
</tr>
<tr>
<td>Selenium</td>
<td>1.8 – 18</td>
<td>BDL – 4.2</td>
<td>0.60 – 4.9</td>
<td>BDL – 0.75</td>
</tr>
<tr>
<td>Thallium</td>
<td>BDL – 45</td>
<td>All BDL</td>
<td>0.1 – 1.8</td>
<td>0.20 – 0.70</td>
</tr>
<tr>
<td>Uranium</td>
<td>BDL – 19</td>
<td>BDL – 16</td>
<td>0.84 – 43</td>
<td>1.2 – 3.9</td>
</tr>
</tbody>
</table>

BDL  Below Detection Limit

Adopted from: EPRI, Coal Ash: Characteristics, Management and Environmental Issues

### 7.4 LEACHING OF TOXIC ELEMENTS FROM COAL ASH

In a recent incident the coal ash spill at the Tennessee Valley Authority’s (TVA) Kingston coal-burning power plant caused a big alarm due to environmental risks involved. The incident became a major subject of investigation of potential environmental and health impacts.

Three major environmental risks were found during this investigation:

1. Release of high levels of fine particle size (<10μm) toxic and radioactive elements.
   
   Toxic elements  As  75 mg/Kg
Radioactive elements $^{226}\text{Ra} + ^{228}\text{Ra}$

150 µg/Kg 

8pCi/g

2. Contamination of surface waters – only in trace levels in Emory and Clinch rivers, due to dilution in the downstream

3. Accumulation of As-rich and Hg-rich coal ash in river sediments

Coal fly ashes are complex particles of a variable composition. The composition of coal fly ash is mainly dependent on the combustion process, the source of coal and the precipitation technique. Toxic constituents in these particles are metals, polycyclic aromatic hydrocarbons and silica. The potential for leaching of these metals not only depends on the total metals content but also influenced by the crystallinity of the fly ash, as this would dictate whether the metals are incorporated within the gaseous phase or within crystalline compounds. The metals in the gaseous phase are expected to leach at much lower rate than that from the crystalline phase. Since the degree of crystallinity is a function of boiler design and remains relatively constant for a given source, leachable materials remain relatively constant for a given ash source. A number of state regulatory agencies have issued source approval for specific generating facilities after the consistency of these materials had been demonstrated. For stabilized soil, the leachability of metals not only depends on the property of the fly ash but also the soil that is used; for example some of these metals leached from the fly ash may to be adsorbed on the clay minerals of the soil.

Experiments conducted on the leaching of metals from the coal combustion ash have revealed that land disposal of coal ash can have potential impact on the ecosystem with increasing acidity of precipitation. It was observed that the toxicity and metal concentrations of the leachates were highest when ash was leached with HCl at pH 4, while the toxicity and concentrations of ash leached with acetic acid (CH$_3$COOH) were significantly lower compared with ash leached with HCl. The toxicity of the aqueous leachates and concentrations of metals-arsenic, cadmium, chromium, copper, iron, lead, nickel, silver, and zinc, were measured using Microtox and atomic absorption spectrometry, respectively. The table below gives the results of these tests as compared to the EPA fresh water acute criteria.
Table 26  Fresh water acute criteria and metals concentrations (µg/L) in coal ash

<table>
<thead>
<tr>
<th>Metal</th>
<th>EPA fresh water acute</th>
<th>HCl</th>
<th>CH₃COOH</th>
</tr>
</thead>
<tbody>
<tr>
<td>As</td>
<td>340</td>
<td>12.3</td>
<td>8.7</td>
</tr>
<tr>
<td>Cd</td>
<td>1.8</td>
<td>26</td>
<td>2</td>
</tr>
<tr>
<td>Cr</td>
<td>16</td>
<td>13.7</td>
<td>3</td>
</tr>
<tr>
<td>Cu</td>
<td>-</td>
<td>277.3</td>
<td>74.3</td>
</tr>
<tr>
<td>Fe</td>
<td>-</td>
<td>518.7</td>
<td>82.3</td>
</tr>
<tr>
<td>Pb</td>
<td>65</td>
<td>30</td>
<td>3</td>
</tr>
<tr>
<td>Ni</td>
<td>470</td>
<td>29</td>
<td>13</td>
</tr>
<tr>
<td>Zn</td>
<td>120</td>
<td>381</td>
<td>214</td>
</tr>
</tbody>
</table>

*Source: EPA fresh water acute criteria*

It is noticed that with HCl at pH 4 concentrations of Cd, Cu, and Zn were higher than the EPA fresh water criteria, while only Cu and Zn were higher when CH₃COOH was used. Low soil pH aides the increase in leachability of metals and the metal availability in soils is altered by change in pH due to addition of coal combustion residues. Increased pH was found to generally reduce the availability of Cd, Cu, Ni, Pb, Zn, and other metals.

### 7.5 UTILIZATION AND DISPOSAL OF COAL ASH

About 45% of coal ash produced in power plants is utilized in many construction and geotechnical purposes. Physical and chemical characteristics of coal ash make it suitable for such useful applications with the primary use of fly ash being as an ingredient in concrete. Bottom ash and coarser boiler slag are utilized as road base materials and for structural fills. Coal ash which is not put to any beneficial use is disposed of and stored in impoundments.

These impoundments or landfills may be located onsite of a power plant or may be sometimes located somewhere outside. These disposal sites are regulated according to the applicable siting requirements, engineering controls, like liners, leachate collection system, run-on and run-off controls etc. The fly ash in these landfills settles to the bottom. In some cases
treatment chemical may be added to improve settling, to control pH, or to remove dissolved constituents. The settles ash solids may be then either left in place or may be dredged out to be put to some beneficial uses as mentioned above.

Coal as disposal sites are so far mostly managed by the State regulations where they are situated. Their design, siting, engineering controls in respect of quality and setup of liners, leachate collection system, gradients, run on and runoff controls have not been up to the federal standards, so has been their groundwater monitoring and corrective action requirements in case of statistically significant increase noticed in ground water pollutants. It has been only lately that regulatory and engineering controls for new or expanded units permitted between 1994 and 2004 had tightened according to a study by US EPA and US Department of Energy (DOE) published in 2006.

The potential environmental impacts of coal ash spills depend on the characteristics of the disposal site, characteristics of the coal ash and FGD wastes, control method and the degree of control employed. In general, the major potential impacts are ground and surface water contamination and the "degradation" of large quantities of land. Because of continued use of coal as primary fossil fuel for power generation, the possibility of significant environmental impacts, both regionally and nationally, exist. Both Federal and privately-funded programs are developing additional data and information on disposal of FGD sludges and coal ash.
CHAPTER 8: MOVING AHEAD WITH INJECTING SODIUM SULFIDE Na₂S 39% AQUEOUS SOLUTION IN FLY ASH CONDITIONING SYSTEM

As mentioned in Chapter 5 item 5.6 above the use of 15 gallons per day aqueous sodium sulfide injection worked satisfactorily well in stabilizing the MSW residue ashes, it was decided to continue with carrying out further experiments with use of this chemical as part of this project.

A general molecular equation for reaction of aqueous sodium sulfide with trace metal, for example with a chloride compound of Cd, is given below:

\[
\text{CdCl}_2 + 2\text{Na}_2\text{S}_{(aq.)} \rightarrow \text{CdS} + 2\text{NaCl}_{(aq.)}
\]

A temporary set up consisting of a 55-gallon drum of aqueous sodium sulfide specially arranged for this purpose and a positive displacement variable speed chemical injection pump with a discharge capacity of 1 gal./hr mounted at the top of the drum was used. Four more tests were conducted using the same temporary set up and their results were added up to develop a table of summary results for one-tailed 90% confidence interval by Student’s t-statistical method.

The results of leachability tests for Cd and Pb carried out during Feb.–March 2016 and a summary of these results from treatment of FGD and Fly ash residues with varying injection rates of 10 - 15 gallons per day aqueous sodium sulfide calculated as per students t-distribution is given in the table below.
The results of the study showed that leachability of all 8 heavy metals was controlled much within the EPA regulatory limit of each. The two generally hard to control heavy metals, e.g. Cd and Pb were controlled at 33.6% and 6.5% leachability limits, respectively.

The leachability control range of Cd (33.6%) and Pb (6.5%) compares very well with what had been achieved by use of dolomitic fine lime that had resulted in control of Cd at 34.1% and that of Pb at 18.1% of leachability limits from the results of 28 sample tests as shown in Table 18 above.

The resulting ash also meets the criteria for corrosivity (pH < 12.5) as well as for ignitability and reactivity. The liquid sulfide mixes with scrubber and APC system FA in very small w/w percentage (<0.1%) of ash to be treated and in a confined atmosphere of FA conditioning screw smoothly and without friction to be ignitable. It is released in so small quantities that it does not present any danger to human health and environment.

The liquid chemical has now been in use at the facility for long period of time and has not shown any of the above hazardous waste conditions.

**Source:** Hampton Steam Plant data
After the use of aqueous sodium sulfide was proved successful in stabilization and treatment of MSW reside ash at Hampton/NASA Steam Plant a permanent set up with two TACMINA make PW series Solenoid-driven Diaphragm Metering pumps connected to the suction of a 165 gallon capacity container was established in April of 2016 at the facility. One pump was to be operated at a time with the other as standby.

The above results of the 5 tests conducted with treating the facility’s reside ash with 15 gallons per day of aqueous sodium sulfide injection were conveyed to the State environmental regulatory authority informing facility’s decision to henceforth convert to use of aqueous sodium sulfide for its residue ash treatment.

The regular use of aqueous sodium sulfide treatment was started on 5/11/2016.

A 1/2” stainless steel discharge pipe was laid out from the pump to fly ash conditioning screw outside. Warm boiler process water was mixed on the side of the chemical discharge line at the fly ash conditioning screw. The chemical and warm boiler process water mix and condition the fly ashes and make it into a slurry which is then conveyed by a rotating screw on to a vibrating conveyor which carries furnace bottom ashes by means an incline conveyor. The treated fly ashes and the bottom ashes following it are conveyed to an ash storage area as combined ash. Each aqueous sodium sulfide injection pump had a full load discharge capacity of over 150 gallon per day at the fly ash conditioning screw as set according to the stroke length and the frequency of the strokes per minute, up to a maximum of 300 strokes/min. A partial stroke frequency setting of 10 - 30 strokes/min gave the desired variation of 8 g/day - 40 g/day chemical discharge at the fly ash conditioning screw.

8.1 RESIDUE ASH CHARACTERIZATION TESTS AND ANALYSIS

In order to establish a general applicability of sodium sulfide treatment chemical several rounds of residue ash testing were planned that will replicate its effectiveness in all the running scenarios of boiler operating processes. These included, but not limited to, following:

1. Flue gas cleaning condition changes:
   These affect the SDA residues characteristics.
   a. Reagent specific gravity range: 1.01 - 1.03
b. SDA outlet (F. F. inlet) temperature range: 375° F - 430° F

2. Particulate (Fly Ash) collection system variables in Fabric Filters:
   a. All 3 modules in service
   b. Only 2 modules in service

3. Boiler running conditions:
   a. **One boiler shutdown**: Any time it is determined to shutdown a boiler, start collecting residue ash sample on hourly basis as soon as possible and complete 8-hour sample collection that will be kept for processing later and lab testing.
   b. **Boiler Startup**: At any time a boiler is starting up, start collecting ash sample as soon as possible after lighting fires, and complete 8-hr ash collection as above.
   c. **Both boilers shutting down**: At any time it is determined to shutdown both boilers for any reason, start collecting samples as soon as possible and complete as many hourly samples as ash is available and seen dropping from shaker pan.
   d. **Both boilers starting up at intervals**: Collect 8-hour sample soon after startup of the first boiler.

4. Sodium sulfide injection rate:
   a. At current injection rate: 15 g/day
   b. Range of Injection rate to be tested: 8 g/day – 40 g/day

5. Any other process variation and decided as warranted:
   a. Boiler steaming rate
   b. Boiler experiencing upset conditions

   A 14-sample residue ash re-characterization schedule was planned with above multivariate conditions.
Samples were collected during various operating conditions of the boilers and at varying chemical injection rates. A total of 15 tests (one additional test over 14 initially planned) were conducted during the period 6/2/2016 through 6/29/2016.

The cumulative results of leachability of 8 heavy metals regulated by EPA obtained from these tests and analyzed using Student’s t-distribution with 90% C.I. one-tailed are tabulated below.

Table 28  Summary of 15 aqueous sodium sulfide treatment tests

<table>
<thead>
<tr>
<th>METAL</th>
<th>AVERAGE mg/L</th>
<th>UCL mg/L</th>
<th>RT mg/L</th>
<th>%RT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>As</td>
<td>0.0200</td>
<td>0.0277</td>
<td>5</td>
</tr>
<tr>
<td>Barium</td>
<td>Ba</td>
<td>0.5568</td>
<td>0.6224</td>
<td>100</td>
</tr>
<tr>
<td>Cadmium</td>
<td>Cd</td>
<td>0.2767</td>
<td>0.3710</td>
<td>1</td>
</tr>
<tr>
<td>Chromium</td>
<td>Cr</td>
<td>0.0358</td>
<td>0.0575</td>
<td>5</td>
</tr>
<tr>
<td>Lead</td>
<td>Pb</td>
<td>0.3951</td>
<td>0.6753</td>
<td>5</td>
</tr>
<tr>
<td>Mercury</td>
<td>Hg</td>
<td>0.0036</td>
<td>0.0080</td>
<td>0.2</td>
</tr>
<tr>
<td>Selenium</td>
<td>Se</td>
<td>0.0822</td>
<td>0.1056</td>
<td>1</td>
</tr>
<tr>
<td>Silver</td>
<td>Ag</td>
<td>0.0100</td>
<td>0.0100</td>
<td>5</td>
</tr>
</tbody>
</table>

15 Sample points for Cadmium and Lead
15 Sample points for all 8 metals

Summary Results show that Residue Ash does not exhibit Toxic Characteristics

Source: Hampton Steam Plant data

The results of the individual tests are also included in the table below.

The results of the study involving 15 samples drawn at varying boiler operating conditions showed that leachability of all 8 heavy metals was controlled much within the EPA regulatory limit of each. The two generally hard to control heavy metals, e.g. Cd and Pb were controlled at 37.1% and 13.5% leachability limits, respectively.

The leachability control range of Cd (37.1%) and Pb (13.5%) compares very well with what had been achieved by use of dolomitic fine lime that had resulted in control of Cd at 34.1%
and that of Pb at 18.1% of leachability limits from the results of 28 sample tests as shown in Table 18 above.

The results in both instances were analyzed by Students t-analysis at one-tailed 90% confidence interval.

Table 29  Results of 15 aqueous sodium sulfide treatment tests

<table>
<thead>
<tr>
<th>Sample No</th>
<th>Sample Date</th>
<th>As</th>
<th>Ba</th>
<th>Cd ≤ 0.026</th>
<th>Cr</th>
<th>Pb ≤ 0.050</th>
<th>Hg</th>
<th>Se</th>
<th>Ag</th>
<th>Entr. Fluid</th>
<th>End Pt pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 HNSP-0616-SST1</td>
<td>6/25/2016</td>
<td>0.005</td>
<td>0.250</td>
<td>0.026</td>
<td>0.10</td>
<td>0.050</td>
<td>0.0002</td>
<td>0.050</td>
<td>0.000</td>
<td>1</td>
<td>11.75 / 8.89</td>
</tr>
<tr>
<td>2 HNSP-0616-SST2</td>
<td>6/26/2016</td>
<td>0.050</td>
<td>0.460</td>
<td>0.110</td>
<td>0.010</td>
<td>0.050</td>
<td>0.0002</td>
<td>0.221</td>
<td>0.000</td>
<td>1</td>
<td>11.22 / 7.45</td>
</tr>
<tr>
<td>3 HNSP-0616-SST3</td>
<td>6/27/2016</td>
<td>0.050</td>
<td>0.557</td>
<td>0.136</td>
<td>0.010</td>
<td>0.065</td>
<td>0.0002</td>
<td>0.203</td>
<td>0.000</td>
<td>1</td>
<td>11.45 / 7.07</td>
</tr>
<tr>
<td>4 HNSP-0616-SST4</td>
<td>6/28/2016</td>
<td>0.050</td>
<td>0.473</td>
<td>0.087</td>
<td>0.010</td>
<td>0.050</td>
<td>0.0002</td>
<td>0.203</td>
<td>0.000</td>
<td>1</td>
<td>11.29 / 8.05</td>
</tr>
<tr>
<td>5 HNSP-0616-SST5</td>
<td>6/29/2016</td>
<td>0.005</td>
<td>0.478</td>
<td>0.368</td>
<td>0.010</td>
<td>0.132</td>
<td>0.0002</td>
<td>0.050</td>
<td>0.000</td>
<td>2</td>
<td>11.62 / 7.83</td>
</tr>
<tr>
<td>6 HNSP-0616-SST6</td>
<td>6/30/2016</td>
<td>0.005</td>
<td>0.602</td>
<td>0.137</td>
<td>0.010</td>
<td>0.050</td>
<td>0.0002</td>
<td>0.050</td>
<td>0.000</td>
<td>1</td>
<td>10.97 / 7.35</td>
</tr>
<tr>
<td>7 HNSP-0616-SST7</td>
<td>6/31/2016</td>
<td>0.005</td>
<td>0.364</td>
<td>0.085</td>
<td>0.010</td>
<td>0.050</td>
<td>0.0002</td>
<td>0.050</td>
<td>0.000</td>
<td>1</td>
<td>11.10 / 7.01</td>
</tr>
<tr>
<td>8 HNSP-0616-SST8</td>
<td>6/3/2016</td>
<td>0.005</td>
<td>0.250</td>
<td>0.085</td>
<td>0.010</td>
<td>0.050</td>
<td>0.0050</td>
<td>0.050</td>
<td>0.000</td>
<td>1</td>
<td>11.00 / 7.30</td>
</tr>
<tr>
<td>9 HNSP-0616-SST9</td>
<td>6/4/2016</td>
<td>0.005</td>
<td>0.708</td>
<td>0.373</td>
<td>0.173</td>
<td>1.350</td>
<td>0.0006</td>
<td>0.050</td>
<td>0.000</td>
<td>2</td>
<td>11.96 / 6.44</td>
</tr>
<tr>
<td>10 HNSP-0616-SST10</td>
<td>6/5/2016</td>
<td>0.005</td>
<td>0.810</td>
<td>0.460</td>
<td>0.202</td>
<td>3.040</td>
<td>0.0004</td>
<td>0.050</td>
<td>0.000</td>
<td>2</td>
<td>11.56 / 5.52</td>
</tr>
<tr>
<td>11 HNSP-0616-SST11</td>
<td>6/6/2016</td>
<td>0.050</td>
<td>0.560</td>
<td>0.836</td>
<td>0.010</td>
<td>0.214</td>
<td>0.0002</td>
<td>0.050</td>
<td>0.000</td>
<td>2</td>
<td>11.43 / 5.36</td>
</tr>
<tr>
<td>12 HNSP-0616-SST12</td>
<td>6/7/2016</td>
<td>0.005</td>
<td>0.535</td>
<td>1.010</td>
<td>0.020</td>
<td>0.241</td>
<td>0.0002</td>
<td>0.050</td>
<td>0.000</td>
<td>2</td>
<td>11.13 / 5.54</td>
</tr>
<tr>
<td>13 HNSP-0616-SST13</td>
<td>6/8/2016</td>
<td>0.005</td>
<td>0.827</td>
<td>0.335</td>
<td>0.032</td>
<td>0.403</td>
<td>0.0002</td>
<td>0.050</td>
<td>0.000</td>
<td>2</td>
<td>10.76 / 5.46</td>
</tr>
<tr>
<td>14 HNSP-0616-SST14</td>
<td>6/9/2016</td>
<td>0.005</td>
<td>0.845</td>
<td>0.253</td>
<td>0.010</td>
<td>0.112</td>
<td>0.0002</td>
<td>0.050</td>
<td>0.000</td>
<td>1</td>
<td>11.75 / 7.39</td>
</tr>
<tr>
<td>15 HNSP-0616-SST15</td>
<td>6/10/2016</td>
<td>0.050</td>
<td>0.614</td>
<td>0.167</td>
<td>0.010</td>
<td>0.050</td>
<td>0.0002</td>
<td>0.050</td>
<td>0.000</td>
<td>2</td>
<td>11.60 / 5.54</td>
</tr>
</tbody>
</table>

Source: Hampton Steam Plant data

All numerical values are leachability in mg/L as tested by EPA Method 1311

A pump setting of 15 strokes per minute adopted for three tests at serial number 2 to 4 above gave a nominal injection rate of 12.5 g/day at the discharge point of chemical at fly ash conditioning screw.

The results indicated a sustained control of both Cd and Pb within the permit limits, except that they include one outlier for leachability of Cd for the sample HNSP-0616-SST12
drawn on 6/28/2016 when the leaching of this trace metal was observed as 1.010 mg/L which is 1% over the EPA limit for this metal.

The results also include an uncharacteristic high result of Pb leaching at 3.040 mg/L as tested for sample HNSP-0616-SST10 drawn on 6/21/2016. Although it was well within the EPA leachability limit for this metal, but this trace metal was not quite often observed leaching at this high ppm value from the dozens of samples tested.

The consumption rate of aqueous sodium sulfide was estimated as below:

**Injection rate at 15 strokes per minute:** 12.5 g/day

**Sp. Density of aqueous sodium sulfide:** 1.12 – 1.13

**Estimated chemical consumption rate:** 120 lb/day

Estimated percentage of chemical use for treatment of fly ash:

Fly ash generated from combustion of 240 tons/day MSW = 12 tons/day

Estimated % of chemical use for trace metal stabilization = 120 lb/24,000 lb ash

\[= 0.5\% \text{ by weight of fly ash}\]

Total ash generated from combustion of 240 tpd of MSW = 80 tons/day

Estimated % of chemical use for treatment of total ash = 120 lb/160,000 lb ash per day

\[= 0.075\% \text{ by weight of total ash}\]

These results again indicate the pH of the treated residue during all tests was below 12 and also proved non-hazardous in respect of ignitability and reactivity.

A regression analysis of these two metals without taking into account these outliers has also been carried out and is shown in the respective charts drawn below.

The linear and exponential regression models of these results are drawn below.
Figure 21  Logarithmic regression models for 15 values of Cd and Pb, drawn together

Figure 22  Logarithmic regression model for all 15 values of Cd only
Figure 23  Logarithmic regression model for all 15 values of Pb only

Figure 24  Linear and Logarithmic regression models for 14 values of Cd excluding outlier
Figure 25  Linear and Logarithmic regression models for 14 values of Pb excluding outlier

Raw data and details of test results are included in Appendix E.
8.2  SETTING UP PERMANENT CHEMICAL INJECTION SYSTEM

Initially a small metering pump was mounted over a 55 gallon plastic drum containing the sodium sulfide aqueous solution as shipped by the vendor as shown below as a temporary experimental set up to start with trial treatment.

Figure 26  Temporary set up for liquid treatment chemical trials

*Source:* Hampton Steam Plant

A Check valve, B Discharge-side joint, C Foot valve

Five trial tests were conducted with this temporary set up. The treatment of FGD and Fly ash residues with varying injection rates of 10 - 15 g/day aqueous sodium sulfide solution proved positive in immobilizing the heavy metals in the combined residue ash when tested with TCLP method in all the five treatment trials runs.

After the trials with temporary chemical injection arrangement were successfully completed it was replaced with a permanent set up with TACMINA make PW series Solenoid-driven Diaphragm Metering pump connected to the suction of a 165 gallon capacity polyethylene tank in April of 2016.
The maximum discharge capacity of the PW-30 R model used was 30 ml/min when set at 300 strokes/min and stroke length set at 100%. The range of setting varies from 0.1 - 300 strokes/min (max). The pump discharges obtained is a pulse type flow.
Figure 31  A PW series Standard pulsing type diaphragm metering pump

The injection rate was to be controlled using one of the following three available options:

1. Setting the discharge capacity by manual operation
2. Setting the discharge capacity by setting the stroke length
3. Controlling operation using signal input

A PWM series Analog-input type pump was chosen (option 2) and chemical flow was set by adjusting the strokes per minute rate keeping the stroke length at 100%. The numerical value of the stroke/min display can be done using the Up and Down arrows.

The product flow was first manually measured using a graduated measuring cylinder and setting the pulsing rate to random strokes per minute, e.g. 15, 20, or 25 while timing the pump’s operation for a set number of minutes, say 5 minutes. The pumps discharge was thus calculated in ml/min, which could then be converted to gal/day rate. An average chemical flow rate in range of 10 to 15 g/day was targeted based on the experimental data that provided the successful immobilization of heavy metals in the facility’s residue ash during full scale testing with both boilers operating at their full rated capacity.

A 15 strokes/min setting provided on an average a chemical flow rate of 33 ml/min which equals to 12.5 g/day when both boilers are in service. Several sets of measurements were done to verify the chemical flow rate per day.
It was also worked out that in case of only boiler in service a chemical discharge rate of 10gal/min was needed to satisfactorily immobilize the heavy metals in the residue ash resulting from the operation of a single boiler. This flow was achieved at the pump pulsing rate of 12 strokes/min, as against the setting of 15 strokes/min to obtain an injection rate of 12.5 g/day.

The control panel of the PWM series analog-input type pump used is show below.

![PWM series pump operation](image)

<table>
<thead>
<tr>
<th>No.</th>
<th>Name</th>
<th>Function</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td>PL Lamp</td>
<td>This lights while power is supplied</td>
</tr>
<tr>
<td></td>
<td></td>
<td>During operation it blinks at timing of operation</td>
</tr>
<tr>
<td>(2)</td>
<td>STOP Lamp</td>
<td>This lights while the pump is shutdown</td>
</tr>
<tr>
<td>(3)</td>
<td>STOP/STAR</td>
<td>This is used to start or stop operation</td>
</tr>
<tr>
<td>(4)</td>
<td>MODE Key</td>
<td>This used to switch the operation mode</td>
</tr>
<tr>
<td>(5)</td>
<td>SEY Key</td>
<td>This is used to enter what has been set</td>
</tr>
<tr>
<td></td>
<td></td>
<td>It is used to transfer from the mode display screen to the setting screens</td>
</tr>
<tr>
<td>(6)</td>
<td>↓↑ KEY</td>
<td>These are used to change the setting values</td>
</tr>
<tr>
<td>(7)</td>
<td>Mode display</td>
<td>The lamp alongside the now operating or now set mode lights</td>
</tr>
<tr>
<td>(8)</td>
<td>SAFE mode display</td>
<td>This lights when the SAFE mode setting is ON</td>
</tr>
<tr>
<td>(9)</td>
<td>ECO LAMP</td>
<td>This lights during operation that involves minimal power consumption.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>*This lights regardless of the ECO mode setting</td>
</tr>
<tr>
<td>(10)</td>
<td>DISPLAY</td>
<td>The setting values are displayed here.</td>
</tr>
</tbody>
</table>

Figure 32  PWM series analog-input pump

Two of such PWM series analog-input pumps were installed. One pump was to be operated at a time with the other was kept as standby.

The permanent sodium sulfide injection system as shown below was installed, tested and completed on 6/17/2016. Initially the chemical discharge from the pump was injected into the boiler process water line that carried the mixture of boiler water and sodium sulfide up to the discharge point at the conditioning screw.
The 15 characterization tests conducted during June 2016 simulated to a considerable extent the actual steam plant operating conditions and some of the tests included the periods when either one or both the boilers were shut down or were starting up.

Other test conditions varied during the 15 characterization tests included:

1. Fabric Filter inlet temperature
2. Fabric Filter modules in service: either all 3 or a pair of 1-2, 2-3, or 1-3
3. Reagent specific gravity: 1.0 to 1.2
4. SO$_2$ control parameters: 40 ppm to 60 ppm
5. Boilers’ output rates were kept constant and steady steaming was ensured during the testing period. However, daily normal on-line cleaning operations of soot blowing made the boilers swing for short durations and thus simulated the normal boiler running conditions to a considerable extent.
6. Boiler upsets that included shutting down and starting up of one or both the boilers during the test period was managed to be included to simulate actual operating scenarios.
The following table includes the variations in operating conditions during that were managed during the 15 tests conducted between 6/1/2016 through 6/29/2016. The results of these 15 tests are shown in Table 30 below.

Table 30  Boilers’ operating condition variations during 15 tests

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Test ID</th>
<th>Test Date</th>
<th>Fabric Filter Inlet Temp. ⁰F</th>
<th>F.F. Modules in service</th>
<th>SO₂ control set point (ppm)</th>
<th>Reagent specific gravity</th>
<th>Sodium Sulfide injection rate (gal/d)</th>
<th>Boilers running/system status</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>HNSP-0616-SST1-A</td>
<td>6/2/2016</td>
<td>375</td>
<td>All 3</td>
<td>50</td>
<td>1.02</td>
<td>12.5</td>
<td>Fly ash system maint.</td>
</tr>
<tr>
<td>2</td>
<td>HNSP-0616-SST 2</td>
<td>6/3/2016</td>
<td>400</td>
<td>All 3</td>
<td>40</td>
<td>1.02</td>
<td>12.5</td>
<td>Normal</td>
</tr>
<tr>
<td>3</td>
<td>HNSP-0616-SST 3</td>
<td>6/6/2016</td>
<td>430</td>
<td>1, 3</td>
<td>60</td>
<td>1.01</td>
<td>12.5</td>
<td>Normal</td>
</tr>
<tr>
<td>4</td>
<td>HNSP-0616-SST 4</td>
<td>6/7/2016</td>
<td>400</td>
<td>1,2</td>
<td>60</td>
<td>1.01</td>
<td>13.5</td>
<td>Normal</td>
</tr>
<tr>
<td>5</td>
<td>HNSP-0616-SST 5</td>
<td>6/8/2016</td>
<td>430</td>
<td>2,3</td>
<td>60</td>
<td>1.01</td>
<td>16.0</td>
<td>Normal</td>
</tr>
<tr>
<td>6</td>
<td>HNSP-0616-SST 6</td>
<td>6/9/2016</td>
<td>430</td>
<td>All 3</td>
<td>50</td>
<td>1.00</td>
<td>18.0</td>
<td>Normal</td>
</tr>
<tr>
<td>7</td>
<td>HNSP-0616-SST 7</td>
<td>6/10/2016</td>
<td>430</td>
<td>1,3</td>
<td>60</td>
<td>1.01</td>
<td>20.0</td>
<td>Normal</td>
</tr>
<tr>
<td>8</td>
<td>HNSP-0616-SST 8</td>
<td>6/13/2016</td>
<td>400</td>
<td>2,3</td>
<td>60</td>
<td>1.00</td>
<td>14.0</td>
<td>Normal</td>
</tr>
<tr>
<td>9</td>
<td>HNSP-0616-SST 9</td>
<td>6/14/2016</td>
<td>430</td>
<td>All 3</td>
<td>60</td>
<td>1.01</td>
<td>14.0</td>
<td>Normal</td>
</tr>
<tr>
<td>10</td>
<td>HNSP-0616-SST 10</td>
<td>6/21/2016</td>
<td>430</td>
<td>All 3</td>
<td>60</td>
<td>1.01</td>
<td>13.0</td>
<td>Both boilers in startup condition</td>
</tr>
<tr>
<td>11</td>
<td>HNSP-0616-SST 11-A</td>
<td>6/22/2016</td>
<td>430</td>
<td>All 3</td>
<td>60</td>
<td>1.01</td>
<td>10.0</td>
<td>Both boilers shutdown and startup quick</td>
</tr>
<tr>
<td>12</td>
<td>HNSP-0616-SST 12</td>
<td>6/28/2016</td>
<td>430</td>
<td>All 3</td>
<td>60</td>
<td>1.01</td>
<td>10.0</td>
<td>Both boilers in startup condition</td>
</tr>
<tr>
<td>13</td>
<td>HNSP-0616-SST 13</td>
<td>6/24/2016</td>
<td>430</td>
<td>All 3</td>
<td>60</td>
<td>1.01</td>
<td>10.0</td>
<td>Both boilers in startup condition</td>
</tr>
<tr>
<td>14</td>
<td>HNSP-0616-SST 14</td>
<td>6/28/2016</td>
<td>430</td>
<td>All 3</td>
<td>60</td>
<td>1.01</td>
<td>10.0</td>
<td>Normal</td>
</tr>
<tr>
<td>15</td>
<td>HNSP-0616-SST 15-A</td>
<td>6/29/2016</td>
<td>430</td>
<td>All 3</td>
<td>60</td>
<td>1.01</td>
<td>10.0</td>
<td>Normal</td>
</tr>
</tbody>
</table>
Later as an additional precaution and to ensure safe discharge of the measured chemical directly into the fly ash at conditioning screw, a separate dedicated stainless steel pipe line was run from the pumps directly to the fly ash conditioning screw.

Figure 35  Direct chemical injection in fly ash  Figure 36  Loading of dolomite eliminated

After a separate dedicated chemical injection line was laid to directly discharge sodium sulfide chemical into the fly ash conditioning screw, 6 additional confirmatory tests beyond the 15 conducted and shown above were conducted during the month of July/August, 2016 keeping the boilers running at set process conditions without changing any control parameters.

The results of 6 confirmatory tests are reproduced below.
Table 31 Results of 6 confirmatory tests conducted during August 2016

<table>
<thead>
<tr>
<th>Point</th>
<th>Sample No.</th>
<th>Sample Date</th>
<th>Laboratory Test results of all 8 heavy metals(mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>As</td>
</tr>
<tr>
<td>16</td>
<td>HNSP-0716-SST16</td>
<td>7/28/2016</td>
<td>0.050</td>
</tr>
<tr>
<td>17</td>
<td>HNSP-0816-SST17</td>
<td>8/5/2016</td>
<td>0.050</td>
</tr>
<tr>
<td>18</td>
<td>HNSP-0816-SST18</td>
<td>8/9/2016</td>
<td>0.050</td>
</tr>
<tr>
<td>19</td>
<td>HNSP-0816SST19A</td>
<td>8/17/2016</td>
<td>0.050</td>
</tr>
<tr>
<td>20</td>
<td>HNSP-0816-SST20</td>
<td>8/25/2016</td>
<td>0.050</td>
</tr>
<tr>
<td>21</td>
<td>HNSP-0816-SST21</td>
<td>8/28/2016</td>
<td>0.050</td>
</tr>
</tbody>
</table>

The analysis of these final results with the boilers steady state running conditions and with an ensured supply of sodium sulfide treatment chemical through a direct discharge pipe line up to the fly ash conditioning screw indicate a firm and constantly reliable response of the chosen chemical to successfully immobilize the heavy metals in the MSW residue.

The table below shows the cumulative results of a total of 21 characterization tests and an improvement in control of leaching of Cd and Pb which is further dropped to 27.3% and 9.7% respectively of their regulatory limits against 37.1% and 13.6% leaching obtained after conducting 15 tests as shown in table 28 above.

These additional tests thus established a very safe and reliable control of leaching of heavy metals in MSW residue ash by the sodium sulfide treatment method even at very low injection rates.
Table 32  Cumulative results of 21 tests with sodium sulfide treatment: 8/2016

<table>
<thead>
<tr>
<th>METAL</th>
<th>AVERAGE mg/L</th>
<th>UCL mg/L</th>
<th>RT mg/L</th>
<th>%RT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>0.0143</td>
<td>0.0202</td>
<td>5</td>
<td>0.4%</td>
</tr>
<tr>
<td>Barium</td>
<td>0.3977</td>
<td>0.4852</td>
<td>100</td>
<td>0.5%</td>
</tr>
<tr>
<td>Cadmium</td>
<td>0.1976</td>
<td>0.2730</td>
<td>1</td>
<td>27.9%</td>
</tr>
<tr>
<td>Chromium</td>
<td>0.0256</td>
<td>0.0414</td>
<td>5</td>
<td>0.8%</td>
</tr>
<tr>
<td>Lead</td>
<td>0.2622</td>
<td>0.4842</td>
<td>5</td>
<td>9.7%</td>
</tr>
<tr>
<td>Mercury</td>
<td>0.0025</td>
<td>0.0057</td>
<td>0.2</td>
<td>2.8%</td>
</tr>
<tr>
<td>Selenium</td>
<td>0.0667</td>
<td>0.0783</td>
<td>1</td>
<td>7.8%</td>
</tr>
<tr>
<td>Silver</td>
<td>0.0671</td>
<td>0.0085</td>
<td>5</td>
<td>0.2%</td>
</tr>
</tbody>
</table>

| 21 Sample points for Cadmium and Lead |
| 21 Sample points for all 8 metals |

Summary Results show that Residue Ash does not exhibit Toxic Characteristics.

Source: Hampton Steam Plant data

The results of TCLP test results of two other MSW waste-to-energy plants, one from Covanta Fairfax, Virginia and the other Wheelabrator, Portsmouth, Virginia are included in Appendix F for comparison purposes.

8.3 COST SAVINGS, RELIABILITY AND EASE OF OPERATION

Use of aqueous sodium sulfide (at injection rate of 12.5 g/day) has resulted in following cost savings by switching over from dolomite treatment in cost of chemical and labor etc.:

A. Costs for chemicals + shipping and labor:

<table>
<thead>
<tr>
<th>Material costs/mo.</th>
<th>Labor/mo.</th>
<th>Total Costs/year</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Dolomite</td>
<td>$16,039.80</td>
<td>$607.00</td>
</tr>
<tr>
<td>2. Sodium Sulfide</td>
<td>$4,535.18</td>
<td>Nil</td>
</tr>
</tbody>
</table>

Savings in cost of chemicals and labor: $145,339.20/yr
B. Savings in maintenance costs (Est., $400/mo.) $4,800/yr

Est. Total annual savings: $150,139.20

Earlier research had resulted in replacing a proprietary chemical that was used since March 2005 to use of dolomite lime during November of 2008. The savings from changeover of chemicals at that time was estimated as $380,854/yr calculated at FY 09 rates. That had resulted in total savings of approximately $2.86 million during the 7 ½ years it had been kept replaced with dolomite.

Further research to find even a better and cheaper substitute for dolomite, during which 4 or 5 alternative chemicals and operating process adjustments were made, tried and tested for long enough periods of time before they had to be given up for lack of sustained good results, has now resulted in an easily injectable and environmentally safer substitute at much lower associated costs for stabilization of our combustion residues before their disposal to landfill. It has lesser chances of spills and lower footprint compared to use of dolomite.

The cumulative savings from these two changeovers in use of chemicals total over $530,000 per year.
CHAPTER 9: CONCLUSION

This study has determined the characteristics of residue ash from municipal solid waste mass burn waste-to-energy plants. The studies conducted by researching various experimental characterization and stabilization technologies for stabilization and rendering the residue ash non-hazardous for disposal to landfill and those observed and analyzed through the applied research designs at the Hampton facility have resulted in the following conclusions:

(1) The toxic heavy metals in the fly ash generated in the municipal solid waste combustion process are effectively stabilized by using any one of treatment chemicals: a proprietary chemical, dolomite, and sodium sulfide. Use of dolomitic lime had resulted in saving the Hampton facility $380,850 per year (at 2009 rates) since 2009.

(2) The boilers running conditions do not have any significant influence on the stabilization and control of leachability concentration below EPA limit of 1 ppm for Cd and limit of 5 ppm for Pb, as their probability values as obtained by statistical analysis was <0.05.

(3) Stabilization by use of sodium sulfide aqueous solution offers advantage over treatment of fly ash by dolomite in that it eliminates the manpower requirement to individually upload bags of dolomite which are currently being obtained from suppliers in 1 ton super sacks due to fact that no storage silo has been built so far to entertain bulk supplies. Changing over to liquid sodium sulfide treatment therefore results in savings in manpower deployment by the facility as well as result in operational ease of pumping a liquid solution to fly ash.

(4) Another effective treatment of fly ash is using complex agents such as Ethylene Diamine Tetra-acetic Acid disodium salt (EDTA). The cost comparison between dolomite and EDTA and also between sodium sulfide aqueous solution and EDAT has not been examined, but it is given that complex agents like EDTA are bound to cost much more than either of the other two treatment chemicals and will go against the very goal of this study, that is to find a cost-effective solution for fly ash treatment at the Hampton facility.
(5) The concentrations of heavy metals especially Cd and Pb in the fly ash collected in scrubber hopper after flue gas scrubbing and those precipitated in fabric filters bags have increased after modification of APC equipment to meet EPA’s new emission guidelines. Very low concentrations of these two pollutants are found emitting through the stack flue gases as has been found out from the results of Hampton facility’s annual stack emission tests during last 8 or 9 years.

(6) Toxic heavy metals Cd and Pb bind themselves less with the finer particles in fly ash as compared to binding with courser ash particles of the bottom ash. The immobilization of Cd and Pb in finer fly ash particles therefore requires additional stabilization products.

(7) The heavy metal studies in municipal waste combustion ash indicate that their behavior is pH dependent. It has been found that the final pH of ash suspension during TCPL testing affects the behavior of retention or release of Cd and Pb and is dependent upon the initial pH of the solution, the alkalinity, and the buffer capacity of the ash.

The strong acidic fluid used during TCLP testing weighing twenty times the weight of ash sample and then tumbled for eighteen hours to simulate the long term landfill disposal conditions has either pH of 4.93 (Fluid 1) or a pH of 2.88 (Fluid 2). As the pH is based on logarithmic scale, Fluid 2 is more than 100 times acidic than Fluid 1 and is called for the residues that contain significant caustic buffers. Determination of which TCLP fluid to use for a non-homogeneous waste like MWC residue ash is very critical toxicity leachate testing.

(8) Over the course of finding a cost-effective treatment for stabilization of residue ash the Hampton facility has affected substantial savings in water usage, energy consumption, and cost-of-lime usage by switching over to dolomite fines and it can expect further cost savings are expected by using sodium sulfide aqueous solution treatment by carrying out flue gas scrubbing at elevated temperatures of up to 430°F to continue with savings in water usage.

(9) The MWC residue ash form mass burn facility at Hampton is very heterogeneous and can be used as soil cover material in landfill as the metals and overs from the residue ash are separated during post-combustion process. The facility’s residue ash is not suitable for
disposal or utilization for road pavement or as a mixing agent with construction material. Residues from Refuse Derived Fuel (RDF) plants however may be found advantageous for such usage after a combination of chemical treatment or stabilization with traditional cement or asphalt solidification as suggested in some studies.

(10) The results of this study can be replicated in other mass burn facilities after testing and validation as they would apply for large mass burn facility-specific residues as the current study was carried out for a very small Class II (less than 250 tons per day) facility at Hampton.
CHAPTER 10: RECOMMENDATIONS

The following recommendations for further work on this topic are made:

(1) It may be possible to reduce the current injection rate of 12 gal./day of sodium sulfide aqueous solution for treatment of residue ash by further experimentation. The amount of chemical injection may need to be tweaked in to obtain repeatability and good control of heavy metal leachability. For example, in case the facility is running at reduced boiler loads resulting in reduction in tonnage of residue ash generated, or in the case of one boiler being shut down for repairs, the chemical injection rate can be modulated or reduced to match with the reduced ash loads.

(2) In case for some reason, although very unlikely, use dolomite is chosen as an alternative to sodium sulfide injection for residue ash treatment some reduction in current injection rate of 2% by weight of fly ash may be achievable with acceptable results.

(3) In place of current use of high calcium hydrated lime slurry in the countercurrent spray tower, either a Magnesium-Enhanced Lime (MEL) with an estimated concentration of 5-8 percent magnesium oxide, or dolomitic lime which is normally 20 percent magnesium oxide, may be used with better results, both for acid absorption in flue gases and as a pH binder in fly ash collected from air pollution control (APC) equipment as it is able to achieve high SO$_2$ removal efficiencies in significantly smaller absorber towers. This product is also recommended for further studies as an alternative to the in-line dolomitic lime injection treatment of combustion fly ash.

(4) In the beginning the solid waste incineration residues were used in construction material and soil conditioner, and now with increased awareness of their hazardous nature and more environmental concerns, these are being treated with more care and then properly disposed of in landfills. Experiments are being conducted and processes are being developed in order to extract precious resources like iron, aluminum, copper, zinc and other metals from these residues. Research in this area should be encouraged so that waste incineration is used both for utilizing its energy potential as well as for recycling metals.
It has been generally agreed that the proportion of mass transfer partitioning of metals in flue gases and those in bottom and APC ashes is not affected by variations in waste input and operating conditions. With rapid advances being used to improve the energy efficiency during MSW combustion in recent years, it is difficult to gather enough information that can throw light on the exact physical and chemical processes taking place in modern state-of-the-art municipal solid waste incinerators. Further research on this will improve our knowledge on the effects of varying operating conditions on partitioning of metal in different waste streams and will be useful both for their effective control as well as for their future reuse.
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EPA, Methodology for Assessing Environmental Releases of and Exposure to Municipal Solid Waste Combustor Residuals, EPA/800/8-91/031, April, 1991


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APPENDICES

APPENDIX A

TCLP METHOD 1311

TOXICITY CHARACTERISTIC LEACHING PROCEDURE


EPA Publication Number 530-R-95-036 of July 1995 provides the purpose, sampling approach and analysis method. The MSW combustion residue is tumbled with twenty times its weight of a strong acid for eighteen hours to simulate long term disposal in a landfill. The extraction fluid used normally is anhydrous acetic or nitric acid with either a pH of 4.93 (called Fluid 1) or a pH of 2.88 (called Fluid 2), depending on the initial pH of the extracted residue.

Highly acidic Fluid 2 is used for wastes containing high levels of caustic buffers.

The following flow diagram provides the steps used in TCLP analysis.
APPENDIX B

STANDARD PROCEDURE FOR COLLECTION OF RESIDUE ASH SAMPLES

SOLID WASTE MANUAL # SW 297

HAMPTON/NASA STEAM MPLANT

Residue Ash Testing Protocol

Residue ash generated at the Facility has consistently shown not to be toxic when tested by the Toxic Characteristic Leaching Procedure. Testing protocol includes full characterization and re-characterization four times annually with a single eight hour composite tested for the eight TCLP metals. Quarterly samples will be tested for the eight metals listed in Table 3.2 of the Virginia Hazardous Waste Management Regulations. Testing is done on the combined residue ash only. Results will be evaluated statistically using the Student’s T normal distribution. Results will be reported to DEQ Tidewater Waste Office, within ninety (90) days of sampling with the following information.

1) Date and place of sampling and analysis
2) The names of individuals doing the sampling and analysis
3) Copy of the completed “Chain of Custody” form
4) Sampling and analytic methods used
5) Results of the analysis
6) Statistical analysis of results and historical data
7) Certification signed by the Steam Plant Manager

Residue Ash Characterization

The waste must demonstrate non-hazardous characteristics to be disposed of as solid waste in accordance with Subtitle D standards. The initial testing will be fourteen samples done over at least a seven (7) day period. Each day samples will be gathered and prepared by the procedures of Method HSP-3A. The test results will be evaluated using a Student’s T distribution at a 90% confidence interval, one tailed. Student’s T distribution is for samples that are small compared to the amount of material being tested, and is specifically designated in the TC Rule.
If the upper bound of the confidence interval is above the regulatory threshold for any substance listed in the Hazardous Waste Management Regulations 40CFR 261.24, Table 1, then the waste fails the toxicity characteristic. Two of the initial fourteen samples will be tested for all species found in the Hazardous Waste Management Regulations 40CFR 261.24, Table 1. The others will be tested for metals only: arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver.

**Method HSP-3A: Residue Ash Composite Sample**

The purpose of this method is to obtain a residue ash sample that is truly representative of the mass of waste disposed during that twenty-four hour period. All residue ash samples will be obtained by quartering and weighing, and then will be reduced in sized to two inches or smaller. A twenty pound three component mass proportioned sample will be prepared for analysis. The sample will be delivered under chain of custody to the analytical laboratory. At the laboratory the sample components will be reduced to three eights of an inch or less. The sub-sample components will then be recombined into a one kilogram mass proportion sample. A Toxic Characteristic Leaching Procedure (TCLP) will then be done on the sample in accordance with SW-846 Method 1311 procedures.

**Sampling Equipment:**

2 1 and 5 gallon plastic buckets with covers and 1 quart zip lock bags.
4 Heavy duty foxtail dust brushes, brooms, and shovels.
2 Wheelbarrows with 2” grid screen box
6 ½ cubic yard bins for weighing
1 Platform scale {+/1 lb.}
* Gloves, dust masks, disposable coveralls, plastic bags
* Hammers, shears, and saws

**Gathering the Sample:**

A sample will be gathered over a six hour period while residue ash is being loaded that was generated over the previous twenty-four hours. Each random grab sample will be one half front load bucket of residue taken from each truck being loaded, set into a sample pile, and then covered. The grab will be flattered then quartered. A single quarter will be selected by random coin toss, and that would then be mixed, flattened and quartered. One quarter will be selected for the sample processing. A second quarter would be used for a second distinct sample if needed, but only a single quality sample is needed. As an alternate samples can be grabbed as per Method HSP-5A.
**Initial Sample Preparation:**

Quality assurance procedures will be followed to ensure a true mass proportioned sample is prepared for testing.

1) Separate all materials by passing through a two inch screen. Large residue components will be segregated into metal and combustibles. All aggregate will be swept off the late pieces back into the aggregate sample.

2) Weigh all sample components in plastic bins with the platform scale. Measure weight to the half-pound and record on a residue sample record sheet.

3) Calculate the mass proportion of the residue ash sample in percent aggregate, percent metal and percent unburns.

4) Take some of the unburns and metal and reduce its size to two inch or less for the sample. Reduce to two inch or less by the following methods.

   a) Five pound hammer from a height of twelve inches.

   b) Scissors for unburned paper or plastic.

   c) Shears for sheet metal and bimetallic cans.

   d) Saws for scrap metal (Collect all shavings and add to the aggregate).

5) Document the weight and description of any material removed from sample.

6) Prepare two 20 pounds composite samples as follows:

   a) Calculate pounds required for a mass proportioned sample by multiplying the component proportion decimal by twenty pounds.

   b) Mix, quarter, and then weigh with the scale to get a representative mass proportioned sample of residue ash aggregate.

   c) Put the residue ash aggregate into a clean five gallon container.

   d) Weigh to get representative sub-samples of the metal and unburns.

   e) Put the metal and the unburns sub-samples in a zip lock bag.

   f) Put both sub-sample containers in the five gallon bucket, cover, and seal with forensic tape.
7) Alternative “aggregate only” samples can be directed for samples. Discard all oversized metal and unburns. Mix, quarter and weigh out two twenty pound samples. One will be analyzed and the other archived.

8) Document data and calculations. Initiate chain of custody form and secure the sample. One or two samples will be prepared and analyzed; one quality sample will be prepared and archived.

Analysis:

Analysis will be done in accordance with the procedures prescribed in EPA SW-846, Method 1311, and the Toxic Characteristic Leaching Procedure. Analysis may be done for eight metals, or for all species found in Table 3.2 of the Virginia Hazardous Waste Management Regulations. Results will be evaluated by the methods of EPA SW-846 and applicable Virginia regulations. Archived samples will be used to repeat and quality check. Results will be evaluated statistically in accordance with the methods outlined in Tables 9.1 and 9.2 of EPA SW-846, Test Methods for Evaluating Solid Waste.

Quality Assurance Plan:

One team member will be designated as the Quality Leader. All container weights, scale operation, sample weight data, and quarter selection will be performed by the Quality Leader. The Steam Plant Engineer will monitor sampling and provide on-site verification of data and calculations. Quality points are specific tasks during the sampling that small errors can cause large procedure bias (see Table IV). These tasks must be given extensive effort, oversight, and review. Specific problems with any quality point should be documented by the Quality Leader. The sampling team will review and discuss quality points prior to testing.
Quality assurance can be maintained only if the integrity of the sample is protected. The residue ash sample Chain of Custody must be documented. At all times the custodian must have the sample secured and under complete control. At any time if the custodian cannot assure the custody and integrity of the sample, it will be invalidated and discarded.

Contract laboratories must be a Virginia certified lab and have a full quality assurance program in accordance with guidelines in SW-846 and ASTM Standards. Analysis methods, data, calculations, and results must have quality assurance review and certification.
Periodically an archived sample will be submitted to the laboratory or to a third party laboratory for quality comparisons. Archived samples will be retained until all results are received and analyzed. However, archived samples will not be analyzed for any species unless the holding times listed in Table V can be met.

<table>
<thead>
<tr>
<th></th>
<th>From: Field Collection To: TCLP Extraction</th>
<th>From: TCLP Extraction To: Prep Extraction</th>
<th>From: Prep Extraction To: Analysis</th>
<th>Total Elapsed Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volatiles</td>
<td>14</td>
<td>NA</td>
<td>14</td>
<td>28</td>
</tr>
<tr>
<td>Semi-Volatiles</td>
<td>14</td>
<td>7</td>
<td>40</td>
<td>61</td>
</tr>
<tr>
<td>Mercury</td>
<td>28</td>
<td>NA</td>
<td>28</td>
<td>56</td>
</tr>
<tr>
<td>Metals</td>
<td>180</td>
<td>NA</td>
<td>180</td>
<td>360</td>
</tr>
</tbody>
</table>

Corrective Action

In the event a single quarterly test result was not characteristic of the results of previous testing the quality control sample would be analyzed. The numerical average of the two samples would be considered the sample test results.

The Steam Plant Manager, or his designee, may prescribe corrective action to ensure the sample is representative of the residue mass being disposed. Any corrective action must be completely documented and reported. Corrective Action may include, but is not limited to the following:

1) Repeating the residue sampling.
2) Testing the archived sample to get an average test result.
3) Invalidate any or all samples due to uncertainties caused by facility operating problems, the testing procedure, or a broken chain of custody.
APPENDIX C

HAMPTON STEAM PLANT AUTHORIZATION LETTER FOR USE OF FACILITY DATA FOR THIS STUDY

Sandeep Kumar, Ph.D.
Assistant Professor
Department of Civil & Environmental Engineering
Kaufman Hall, Room 137C, Old Dominion University
Norfolk, VA 23529-0241

27 May 2016

Dr. Kumer,

I understand that Anil Mehrotra, the Plant Engineer here at the Hampton/NASA Steam Plant, is pursuing his Doctorate in Engineering. Your e-mail of 11 May reports that his topic revolves around 'INERTIZATION, UTILIZATION, AND SAFE DISPOSAL OF INCINERATION RESIDUES' and you asked whether he could use the data he collected from the Steam Plant in his dissertation. The Steam Plant commissioned him, and your team, to discover a suitable alternative to the second residue ash treatment of dolomitic lime in Project #200198-010. Further, the Steam Plant assisted with his studies via an education assistance subsidy. With that in mind, and considering we are a public entity seeking a solution to save public funds, I think it is wholly proper for him to use the data he so diligently collected to aid his academic advancement and maximize that investment.

Please let me know if your office requires any further documentation and thank you for your work so far.

Sincerely,

[Signature]

John MacDonald
Plant Manager
Hampton/NASA Steam Plant

Hampton/NASA Steam Plant
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
APPENDIX D

RAW DATA: DOLOMITE TREATMENT METHOD

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Hampton/NASA Steam Plant

Summary of Quarterly Test Results since 2009

Table includes residue ash tests results ending 12/2016

<table>
<thead>
<tr>
<th>METAL</th>
<th>AVERAGE mg/L</th>
<th>UCL mg/L</th>
<th>RT mg/L</th>
<th>%RT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>As 0.0165</td>
<td>0.0218</td>
<td>5</td>
<td>0.4%</td>
</tr>
<tr>
<td>Barium</td>
<td>Ba 0.0136</td>
<td>0.0313</td>
<td>100</td>
<td>0.0%</td>
</tr>
<tr>
<td>Cadmium</td>
<td>Cd 0.3310</td>
<td>0.3650</td>
<td>1</td>
<td>38.5%</td>
</tr>
<tr>
<td>Chromium</td>
<td>Cr 0.0544</td>
<td>0.0804</td>
<td>5</td>
<td>1.6%</td>
</tr>
<tr>
<td>Lead</td>
<td>Pb 0.5419</td>
<td>0.8065</td>
<td>5</td>
<td>16.1%</td>
</tr>
<tr>
<td>Mercury</td>
<td>Hg 0.0028</td>
<td>0.0033</td>
<td>0.2</td>
<td>1.7%</td>
</tr>
<tr>
<td>Selenium</td>
<td>Se 0.0113</td>
<td>0.0148</td>
<td>1</td>
<td>1.6%</td>
</tr>
<tr>
<td>Silver</td>
<td>Ag 0.0022</td>
<td>0.0029</td>
<td>5</td>
<td>0.1%</td>
</tr>
</tbody>
</table>

32 Sample points for all 8 metals

Summary Results show that the Residue Ash does not exhibit Toxic Characteristics
120

Results of 32 Residue Sample Tests with dolomite lime treatment


APPENDIX E

RAW DATA: SODIUM SULFIDE TREATMENT METHOD

June 2016 – August 2016
Hampton/NASA Steam Plant

Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0616–SST1

Sample Collection: Wed June 1, 2016
QA Sample Preparation: Thu June 2, 2016

An 8-hour **residue ash sample collection** with Sodium Sulfide injection is to start at 09:00 AM on Wed, June 1, 2016 & will continue until 04:00 PM Wed. June 1, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to the following schedule and Quality Assurance procedures:

**Wed. June 1:**

**AM Team**
Ensure all test conditions given in Test Day Control Parameters below are set accordingly. **Mention specific existing condition.**
Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

**Starting:**
09:00 AM
Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

**Ending:**
16:00 PM
Continue 1-minute residue ash grab every hour as per above procedure for 8-hours.
Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity: 1.02</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.: 375°F</td>
</tr>
<tr>
<td>Boilers steam rate: 33 pph</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>

**Thu. June 2**

07:30–10:00 AM
Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample. Follow QA and Chain of Custody procedures.

Thank you,

Anil Mehrotra
Plant Engineer
# REPORT OF ANALYSIS

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1105</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>&lt; 0.250</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1105</td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.028</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1105</td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1105</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1105</td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt; 0.0002</td>
<td>mg/L</td>
<td>TLG 06/17/16 1158</td>
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<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.050</td>
<td>1</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1105</td>
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<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
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<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1105</td>
</tr>
</tbody>
</table>

---

James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498  
VELAP# 460013  
EPA# VA00015  
Page 1 of 2
REPORT OF ANALYSIS

NOTES:
JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.
Reproduction of this report is not permitted, except in full, without written approval from James R. Reed & Associates.
The results on this report relate only to the sample(s) provided for analysis.
Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.75
Extraction Fluid used: #1
Final (end point) pH of Extraction Fluid: 9.89

Authorized By: Elaine Claiborne, Laboratory Director
Date: 17-Jun-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax: (757) 873-1498
VELAP# 460013
EPA# VA00015
# ANALYSIS SAMPLE

**CHAIN OF CUSTODY**

**SAMPLE NUMBER:** HSP-0616-1574

**MATERIAL:** Resin AM4-2.5

**SAMPLE PREPARED BY:** Leonard

**METHOD:** HSP-3A

<table>
<thead>
<tr>
<th>CUSTODIAN</th>
<th>FROM</th>
<th>TO</th>
</tr>
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<tbody>
<tr>
<td>Bill Leonard</td>
<td>2 June 16</td>
<td>2 June 16</td>
</tr>
<tr>
<td></td>
<td>275°F</td>
<td>0930</td>
</tr>
<tr>
<td>June Brown</td>
<td>6/2/16</td>
<td>1150</td>
</tr>
<tr>
<td></td>
<td>0930-15 June</td>
<td>1150</td>
</tr>
<tr>
<td></td>
<td>6/15/16</td>
<td>1230</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1215</td>
</tr>
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</table>

Q. A. Weight: 25.5 lb

Test: TOLEP- All Metals

Reviewed By: [Signature]

Date: 6/19/16

Hampton/NASA Steam Plant
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Sample Date:</th>
<th>Method Used:</th>
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<tbody>
<tr>
<td>HSP-0616-5511</td>
<td>6/2/16</td>
<td>HSP 3A</td>
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<table>
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<th>Sample Certified Valid:</th>
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<td>6/11/15</td>
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<td>1003</td>
<td>900</td>
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<td>1106</td>
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<td>260</td>
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<tr>
<td>8</td>
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<td>1000</td>
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<table>
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<td></td>
</tr>
<tr>
<td>24</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### WEIGHT TALLY SHEET (+ or - ½ Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>-4 4 1/2 MB</td>
<td>40 1/2</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>21 5 /</td>
<td>41 1/2</td>
<td></td>
</tr>
<tr>
<td>27</td>
<td>35</td>
<td></td>
</tr>
<tr>
<td>28</td>
<td>38 1/2</td>
<td></td>
</tr>
<tr>
<td>2 5 1/2</td>
<td>35</td>
<td></td>
</tr>
<tr>
<td>41 1/2</td>
<td>28 1/2</td>
<td></td>
</tr>
<tr>
<td>47</td>
<td></td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>52 1/2</td>
<td>23</td>
<td></td>
</tr>
<tr>
<td>5 6 1/2</td>
<td>24</td>
<td></td>
</tr>
<tr>
<td>5 7 1/2</td>
<td>25</td>
<td></td>
</tr>
<tr>
<td>4 6 1/2</td>
<td>26</td>
<td></td>
</tr>
<tr>
<td>4 9 1/2</td>
<td>27</td>
<td></td>
</tr>
<tr>
<td>5 9 1/2</td>
<td>28</td>
<td></td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample # HSP-0616 - SST 1

<table>
<thead>
<tr>
<th>Wet Trench pH: 8.9</th>
<th>Boilers Operating: 1, 2</th>
<th>Weather: cloudy/misty</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Treatment/ Injection Rate: 12.5 gals a day</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Comments:
- Reagent Spec. Grav. 1.02
- Fabric Filter inlet 375
- Boiler steam rate 33 pph
- SO₂ cont. 50 ppm
- FF modules on all 3

Team(s): A - B Teams

Quality Leader: Name Jack Dye, Designation: AO

Team Leader: Name Martin Benfield, Designation: OE

<table>
<thead>
<tr>
<th>Mass Proportion Samples</th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>14.5</td>
<td>73.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unburnt</td>
<td>3.0</td>
<td>25</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unburnable</td>
<td>0.5</td>
<td>1.7</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: Benfield
Date: 6/2/116
Hampton/NASA Steam Plant

Residue Ash Sample Treated with 12.5 gal/day Sod. Sulfide

Method HSP 3A

Sample # HSP- 0616-SST1 (Both boilers in service)

Date: June 2, 2016 (FF inlet Temp. 375° F, SO2 Control 50 ppm)

<table>
<thead>
<tr>
<th></th>
<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>752.5</td>
<td>21.5</td>
<td>44.5</td>
<td>17.5</td>
</tr>
<tr>
<td>Unburnt</td>
<td>257.0</td>
<td>27.0</td>
<td>36.5</td>
<td></td>
</tr>
<tr>
<td>Metals</td>
<td>17.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>1027.0</strong></td>
<td>28.0</td>
<td>46.5</td>
<td></td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td></td>
<td>25.5</td>
<td>49.5</td>
<td></td>
</tr>
</tbody>
</table>

**Mass Proportion in %**

<table>
<thead>
<tr>
<th></th>
<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>73.3%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unburnt</td>
<td>25.0%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Metals</td>
<td>1.7%</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>100.0%</strong></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Mass Proportion in lbs. for 20 lb. sample**

Enter 40.5

**Rounded Up Values in lb.**

<table>
<thead>
<tr>
<th></th>
<th>Calculated</th>
<th>Proportion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>14.5</td>
<td>35.0</td>
</tr>
<tr>
<td>Unburnt</td>
<td>5.0</td>
<td>35.0</td>
</tr>
<tr>
<td>Metals</td>
<td>0.5</td>
<td>28.5</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>20.0</strong></td>
<td><strong>20.00 lbs.</strong></td>
</tr>
</tbody>
</table>
Hampton/NASA Steam Plant

Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0616–SST2

Sample Collection: Thu. June 2, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon Thu. June 2, 2016 & will continue until 19:00 hrs Thu. June 2, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to the following schedule and Quality Assurance procedures:

Thu. June 2:

AM Team

Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Starting:

12:00 noon

Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Ending:

19:00 hrs

Continue 1-minute residue ash grab every hour as per above procedure for 8-hours.

Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (Pre-set on Wed. June 1 at 5:00 PM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>

Fri. June 3

07:30–10:00 AM Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample. Follow QA and Chain of Custody procedures.

Thank you,

Anil Mehrotra
Plant Engineer
<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Toxic Characteristic Leaching Procedure by SW-846 Method 1311</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1607</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.467</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1334</td>
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<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.110</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1334</td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1334</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1334</td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt; 0.0002</td>
<td>mg/L</td>
<td>TLG 06/10/16 1412</td>
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<tr>
<td>Selenium</td>
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<td>6010C</td>
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<td>1</td>
<td>0.221</td>
<td>mg/L</td>
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<td>Silver</td>
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<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1334</td>
</tr>
</tbody>
</table>
**ANALYSIS SAMPLE CHAIN OF CUSTODY**

**SAMPLE NUMBER:** HSP-0611-5577

**MATERIAL:** Restoux Ash

**SAMPLE PREPARED BY:** David H. Weisgerb

**METHOD:** HSP-31

<table>
<thead>
<tr>
<th>CUSTODIAN</th>
<th>FROM</th>
<th>TO</th>
</tr>
</thead>
<tbody>
<tr>
<td>David A. Weisgerb</td>
<td>3 June 16</td>
<td>6-7-16</td>
</tr>
<tr>
<td>Bob Leonard</td>
<td>3 June 16</td>
<td>6-7-16</td>
</tr>
<tr>
<td>Austin Marshall</td>
<td></td>
<td>6-7-16</td>
</tr>
<tr>
<td>Andrew Marshall</td>
<td></td>
<td>6-7-16</td>
</tr>
</tbody>
</table>

Q.A. Weight: **22.5 lb**

Test: **TCLP - All Metals**

Reviewed By: [Signature]

Date: 6/3/2016

Hampton/NASA Steam Plant
50 Wythes Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

<table>
<thead>
<tr>
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<th>Sample Date: 11/8/16</th>
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<tr>
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<td>HSP-3A</td>
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Sample Certified Valid: 11/16/16

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<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
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<tbody>
<tr>
<td>Time</td>
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<td>1:04</td>
<td>1:05</td>
<td>1:00</td>
<td>1:00</td>
<td>1:02</td>
<td>1:05</td>
<td></td>
</tr>
<tr>
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<td>120</td>
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<td>700</td>
<td>700</td>
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<table>
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</thead>
<tbody>
<tr>
<td>Time</td>
<td>12:00</td>
<td>1:04</td>
<td>1:05</td>
<td>1:00</td>
<td>1:00</td>
<td>1:02</td>
<td>1:05</td>
<td></td>
</tr>
<tr>
<td>Initials</td>
<td>120</td>
<td>700</td>
<td>700</td>
<td>700</td>
<td>700</td>
<td>700</td>
<td>700</td>
<td></td>
</tr>
</tbody>
</table>

### WEIGHT TALLY SHEET (+ or − ½ Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 40 lbs</td>
<td>15. 31.5 lbs</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 46 lbs</td>
<td>16. 29 lbs</td>
<td>1. 45 lbs</td>
</tr>
<tr>
<td>3. 47 lbs</td>
<td>17. 45.5 lbs</td>
<td>2. 35 lbs</td>
</tr>
<tr>
<td>4. 45 lbs</td>
<td>18. 42.5 lbs</td>
<td>3. 36 lbs</td>
</tr>
<tr>
<td>5. 53.5 lbs</td>
<td>19. 40.5 lbs</td>
<td>4. 34 lbs</td>
</tr>
<tr>
<td>6. 42.5 lbs</td>
<td>20. 38.5 lbs</td>
<td>5. 39 lbs</td>
</tr>
<tr>
<td>7. 36 lbs</td>
<td>21. 39 lbs</td>
<td>6. 39.5 lbs</td>
</tr>
<tr>
<td>8. 39 lbs</td>
<td>22.</td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>9. 41.5 lbs</td>
<td>23.</td>
<td>1. 10.5 lbs</td>
</tr>
<tr>
<td>10. 31.5 lbs</td>
<td>24.</td>
<td>2. 12.5 lbs</td>
</tr>
<tr>
<td>11. 40.5 lbs</td>
<td>25.</td>
<td>3.</td>
</tr>
<tr>
<td>12. 38 lbs</td>
<td>26.</td>
<td>4.</td>
</tr>
<tr>
<td>13. 31 lbs</td>
<td>27.</td>
<td>5.</td>
</tr>
<tr>
<td>14. 37.5 lbs</td>
<td>28.</td>
<td>6.</td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample # H3P-0416-SS12

<table>
<thead>
<tr>
<th>Wet Trench pH:</th>
<th>8.8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boilers Operating:</td>
<td>Burn</td>
</tr>
<tr>
<td>Weather:</td>
<td>Cloudy+Light Rain</td>
</tr>
</tbody>
</table>

Chemical Treatment/Injection Rate: Sodium Sulfide Injection @ 50 lbs per day

Comments:

---

Team(s): Don Woodman, Jacks Dye, R. Lake, P. Smith
R. Doss, J. Davis

Quality Leader: Name Jacks Dye Designation: Ash Handler

Team Leader: Name Don Woodman Designation: OP. BXC

Mass Proportion Samples

<table>
<thead>
<tr>
<th></th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>797</td>
<td>71.6</td>
<td>14.5</td>
<td>Total: 22.5 Lbs. With bucket and lid.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>292.5</td>
<td>23.6</td>
<td>5.0</td>
<td></td>
</tr>
<tr>
<td>Unburnable</td>
<td>23</td>
<td>2.1</td>
<td>1</td>
<td></td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: [Signature]

Date: 3 June 14
SAMPLE #1SP-0616-5572, DATE 6/3/2016

1) a) aggregate 797
   b) unburnts 292.5
   c) metals 23
   d) total 1112.5

2) a) aggregate \( \frac{a}{d} \times 100 \% = \frac{797}{1112.5} \times 100 \% = 71.6 \%
   b) unburnts \( \frac{b}{d} \times 100 \% = \frac{292.5}{1112.5} \times 100 \% = 26.3 \%
   c) metals \( \frac{c}{d} \times 100 \% = \frac{23}{1112.5} \times 100 \% = 2.1 \%
   d) total = 100 \%

3) a) aggregate 14.2
   b) unburnts 5.2
   c) metals 0.4
   d) total = 20 lbs

4) a) aggregate = 14.5
   b) unburnts = 5.0
   c) metals = 0.5
   d) total = 20 lbs
Hampton/NASA Steam Plant

Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP-0616--SST3

Sample Collection: Fri. June 3, 2016
QA Sample Preparation: Mon. June 6, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon Fri. June 3, 2016 & will continue until 19:00 hrs Fri. June 3, 2016.
The sample will be gathered, prepared, and sealed for analysis as per Method HSP-3A according to the following schedule and Quality Assurance procedures:

Fri. June 3:
AM Team
Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition.
Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Starting:
12:00 noon
Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Ending:
19:00 hrs
Continue 1-minute residue ash grab every hour as per above procedure for 8-hours.
Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (Pre-set on Thu. June 2 at 8:00 PM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
<tr>
<td>3. Ash conveyor issues</td>
</tr>
</tbody>
</table>

Mon. June 6
07:30–10:00 AM Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.
Follow QA and Chain of Custody procedures.

Thank you,

Anil Mehrotra
Plant Engineer
### Toxic Characteristic Leaching Procedure by SW-846 Method 1311

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyte/Data/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1602</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.557</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1330</td>
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<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.136</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1330</td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1330</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>0.065</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1330</td>
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<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt; 0.0002</td>
<td>mg/L</td>
<td>TLG 06/10/16 1410</td>
</tr>
<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.050</td>
<td>1</td>
<td>0.203</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1602</td>
</tr>
<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1330</td>
</tr>
</tbody>
</table>
NOTES:

RA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.

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The results on this report relate only to the sample(s) provided for analysis.

Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.45

Extraction Fluid used: #1

Final (end point) pH of Extraction Fluid: 7.67

Authorized By: (Signature)

Elaine Claiborne, Laboratory Director

Date: 10-Jun-16
ANALYSIS SAMPLE
CHAIN OF CUSTODY

SAMPLE NUMBER: HSP-0616-SS73
MATERIAL:
Residue 6th

SAMPLE PREPARED BY: Tony Taylor
METHOD:
HSP-3A

<table>
<thead>
<tr>
<th>Custodian</th>
<th>From</th>
<th>To</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tony Taylor</td>
<td>6/6/16</td>
<td>0700</td>
<td>6/6/16</td>
</tr>
<tr>
<td>Bill Leonard</td>
<td>6/6/16</td>
<td>1015</td>
<td>6/7/16</td>
</tr>
<tr>
<td>Andrea Marshall</td>
<td>6/7/16</td>
<td>1430</td>
<td>6/7/16</td>
</tr>
<tr>
<td>Andrea Marshall</td>
<td>6/7/16</td>
<td>1500</td>
<td></td>
</tr>
</tbody>
</table>

Q.A. Weight: 22.5
Test: TCLP, all metals

Reviewed By: [Signature]
Date: 6/7/16

Hampton/NASA Steam Plant
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

<table>
<thead>
<tr>
<th>Sample #</th>
<th>HSP-0616-5873</th>
<th>Sample Date:</th>
<th>6/6/2016</th>
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<tbody>
<tr>
<td>Method Used</td>
<td>HSP-3A</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

| Sample Certified Valid: |   |

<table>
<thead>
<tr>
<th>Grab</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
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<tbody>
<tr>
<td>Time</td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
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<td>Initials</td>
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<td></td>
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</tbody>
</table>

<table>
<thead>
<tr>
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<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
<th>13</th>
<th>14</th>
<th>15</th>
<th>16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Initials</td>
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<td></td>
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<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Grab</th>
<th>17</th>
<th>18</th>
<th>19</th>
<th>20</th>
<th>21</th>
<th>22</th>
<th>23</th>
<th>24</th>
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<tbody>
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<td>Time</td>
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<tr>
<td>Initials</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### WEIGHT TALLY SHEET (+ or - ½ Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 53.0</td>
<td>15. 44.10</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 64.0</td>
<td>16. 41.0</td>
<td>1. 33.5</td>
</tr>
<tr>
<td>3. 63.5</td>
<td>17. 45.0</td>
<td>2. 36.0</td>
</tr>
<tr>
<td>4. 70.5</td>
<td>18. 43.5</td>
<td>3. 29.5</td>
</tr>
<tr>
<td>5. 69.0</td>
<td>19. 39.0</td>
<td>4. 50.0</td>
</tr>
<tr>
<td>6. 66.5</td>
<td>20. 37.5</td>
<td>5. 43.0</td>
</tr>
<tr>
<td>7. 67.5</td>
<td>21. 46.6</td>
<td>6. 49.5</td>
</tr>
<tr>
<td>8. 61.0</td>
<td>22. 47.0</td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>9. 57.0</td>
<td>23. 42.5</td>
<td>1. 24.5</td>
</tr>
<tr>
<td>10. 65.5</td>
<td>24. 38.5</td>
<td>2. 11.5</td>
</tr>
<tr>
<td>11. 59.5</td>
<td>25.</td>
<td>3.</td>
</tr>
<tr>
<td>12.</td>
<td>26.</td>
<td>4.</td>
</tr>
<tr>
<td>13. 67.5</td>
<td>27.</td>
<td>5.</td>
</tr>
<tr>
<td>14. 49.0</td>
<td>28.</td>
<td>6.</td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample # HSP-0616-SS13

Wet Trench pH: 9.0  Boilers Operating: 1+2  Weather: Clear

Chemical Treatment/Injection Rate: Sodium Sulfide: 27.5 gal/day

Comments: Test day control parameters: Reagent specific gravity 1.01, F.F. in let temp: -420°F, boiler steam rate - 33 kpl, boiler running condition - Normal, 50% control - 60 ppm, F.F. modulus on - 1+3, Sodium sulfide rate - 3.5 gal per day.

Team (s): D-Team

Quality Leader: Name Tony Taylor  Designation: HSP (OE)

Team Leader: Name  Designation:

<table>
<thead>
<tr>
<th>Mass Proportion Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lbs.</td>
</tr>
<tr>
<td>------</td>
</tr>
<tr>
<td>Aggregate</td>
</tr>
<tr>
<td>Unburnt</td>
</tr>
<tr>
<td>Unburnable</td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: 
Date: 6/6/16
Sample #: HST-0616-5513
Date #: 6/6/2016

1) a) aggregate 12.77
   b) unburnt 282.5
   c) metal 3.6
   d) total 1595.5

2) a) aggregate \( \frac{9}{10} \times 100 \% = 80.8\% \)
   b) unburnt \( \frac{9}{10} \times 100 \% = 17.7 \% \)
   c) metal \( \frac{9}{10} \times 100 \% = 2.2\% \)
   d) total 100 \%

3) a) aggregate 16 lb
   b) unburnt 3.5 lb
   c) metal 15
   d) total 20 lb
Hampton/NASA Steam Plant

Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0616–SST4

Sample Collection: Mon. June 6, 2016
QA Sample Preparation: Tue. June 7, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon Mon. June 6, 2016 & will continue until 19:00 hrs Tue. June 7, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to the following schedule and Quality Assurance procedures:

Mon. June 6:
AM Team

Starting:
12:00 noon

Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Ending:
19:00 hrs

Continuing 1-minute residue ash grab every hour as per above procedure for 8-hours. Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>


Tue. June 7
07:30–10:00 AM
Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample. Follow QA and Chain of Custody procedures.

Thank you,

Anil Mehrotra
Plant Engineer
CLIENT: Hampton/NASA Steam Plant  
ATTN: Anil Mehrotra, Plant Engineer  
ADDRESS: 50 Wythe Creek Road  
CITY: Hampton, VA 23666  
PHONE: (757) 865-1914  
FAX: e: amehrotra@hampton.gov  

SPECIAL NOTES:  

SAMPLE ID: HSP-0616-SST4  
SAMPLE NO: 16-10083  

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>1</td>
<td>&lt;0.050</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1558</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.473</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1325</td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.067</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1325</td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt;0.010</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1325</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt;0.050</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1325</td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7476A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt;0.0002</td>
<td>mg/L</td>
<td>TLG 06/10/16 1407</td>
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<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.050</td>
<td>1</td>
<td>0.299</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1558</td>
</tr>
<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt;0.010</td>
<td>mg/L</td>
<td>PEJ 06/10/16 1325</td>
</tr>
</tbody>
</table>
NOTES:

IRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.

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The results on this report relate only to the sample(s) provided for analysis.

Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.29
Extraction Fluid used: #1
Final (end point) pH of Extraction Fluid: 4.05

Authorized By: [Signature]
Elaine Claiborne, Laboratory Director
Date: 10-Jun-16
**ANALYSIS SAMPLE CHAIN OF CUSTODY**

**SAMPLE NUMBER:** HSP-0616-5574  
**MATERIAL:** Resinoue  
**METHOD:** HSP-3.4

<table>
<thead>
<tr>
<th>CUSTOMIAN</th>
<th>FROM</th>
<th>TO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jack Dye</td>
<td>07/16</td>
<td>09/10</td>
</tr>
<tr>
<td>Bilo Lainord</td>
<td>09/10</td>
<td>07/16</td>
</tr>
<tr>
<td>[Signature]</td>
<td>07/16</td>
<td>6-7/16</td>
</tr>
<tr>
<td>[Signature]</td>
<td>6-7/16</td>
<td>1500</td>
</tr>
<tr>
<td>[Signature]</td>
<td>6-7/16</td>
<td>1500</td>
</tr>
</tbody>
</table>

**Q. A. Weight:** 22.5

**Test:** TCLP - All Metals

Reviewed By: [Signature]  
Date: 6/7/16

Hampton/NASA Steam Plant  
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
<table>
<thead>
<tr>
<th>SAMPLE #</th>
<th>Sample Date</th>
<th>Method Used</th>
</tr>
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<tbody>
<tr>
<td>HSP-0646-5574</td>
<td>7-Jun-16</td>
<td>HSP-3A</td>
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</tbody>
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<tbody>
<tr>
<td>Time</td>
<td>12:07</td>
<td>13:05</td>
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<td>15:15</td>
<td>16:10</td>
<td>17:02</td>
<td>17:47</td>
<td>18:51</td>
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<td>520</td>
<td>002</td>
<td>360</td>
<td>003</td>
<td>012</td>
<td>021</td>
<td>013</td>
<td>015</td>
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<td>12</td>
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<td>16</td>
</tr>
<tr>
<td>Time</td>
<td>17</td>
<td>18</td>
<td>19</td>
<td>20</td>
<td>21</td>
<td>22</td>
<td>23</td>
<td>24</td>
</tr>
<tr>
<td>Initials</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

**WEIGHT TALLY SHEET (+ or - 1/2 Lb)**

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 49</td>
<td></td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 50</td>
<td></td>
<td>1. 56.3</td>
</tr>
<tr>
<td>3. 50.5</td>
<td></td>
<td>2. 35.5</td>
</tr>
<tr>
<td>4. 41</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5. 38</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6. 43</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7. 34</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8. 35.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9. 30.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10. 41</td>
<td></td>
<td></td>
</tr>
<tr>
<td>11. 45</td>
<td></td>
<td></td>
</tr>
<tr>
<td>12. 37</td>
<td></td>
<td></td>
</tr>
<tr>
<td>13. 41</td>
<td></td>
<td></td>
</tr>
<tr>
<td>14. 18.5</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Unburnable (Metal)

1. 12 45

8.5 pH
Residue ash Sample Data Records
(Please Sign and Date below)

Sample # HSP-0616- SST H

<table>
<thead>
<tr>
<th>Wet Trench pH: 8.5</th>
<th>Boilers Operating: 1+2</th>
<th>Weather: Wet/Rainy/Remo</th>
</tr>
</thead>
</table>

Chemical Treatment/ Injection Rate: 13.5 g/lay

Comments:
- Reactor Spec. Grav.: 1.01
- Basic Flue Inlet Temp.: 490°F
- Boilers Steam Rate: 33K ph
- SO2 Control: 60 ppm
- RF Mod: 1/2
- Sodium Sulfide Rate: 3.5 gallon/per/day

Team (s): B

Quality Leader: Name JACK D DYE, Designation: __________

Team Leader: Name BILL LEONARD, Designation: __________

<table>
<thead>
<tr>
<th>Mass Proportion Samples</th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>577</td>
<td>84.7</td>
<td>17.6</td>
<td></td>
</tr>
<tr>
<td>Unburnt</td>
<td>92</td>
<td>13.5</td>
<td>2.5</td>
<td></td>
</tr>
<tr>
<td>Unburnable</td>
<td>12</td>
<td>1.7</td>
<td>0.5</td>
<td></td>
</tr>
</tbody>
</table>

Total: 22.5 Lbs. With bucket and lid.

Verification by Quality Leader or Team Leader

Sign: BILL LEONARD
Date: 6/7/16
Calculation Sheet

Sample #: HSP-0616-5774
Date: 6/7/16

1. Sub Total (lbs)
   a. Aggregate 577.0
   b. Unburnt 92.0
   c. Metal 12.0
   d. Total 681

2. Mass Proportion (%)
   a. Aggregate \( \frac{a}{d} \times 100\% = 84.7 \%
   b. Unburnt \( \frac{b}{d} \times 100\% = 13.5 \%
   c. Metal \( \frac{c}{d} \times 100\% = 1.7 \%
   d. Total 100\%

3. Calculate 20 lb Proportion (lb)
   (upto 2 decimal places)
   a. Aggregate \( A \times 20\text{lb} \) = 11.94
   b. Unburnt \( B \times 20\text{lb} \) = 2.7
   c. Metal \( C \times 20\text{lb} \) = 3.0
   d. Total 20 lb

4. Rounded Up/down (lb)
   (to 1/2 lb)
   a. Aggregate = 17.0 lb
   b. Unburnt = 2.5 lb
   c. Metal = 3 lb
   d. Metal = 20 lb
Hampton/NASA Steam Plant

Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP-0616-SST5

Sample Collection: Tue. June 7, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon Tue. June 7, 2016 & will continue until 19:00 hrs Wed. June 8, 2016.
The sample will be gathered, prepared, and sealed for analysis as per Method HSP-3A according to the following schedule and Quality Assurance procedures:

Tue. June 7:
AM Team

Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition.
Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Starting:
12:00 noon

Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Ending:
19:00 hrs

Continue 1-minute residue ash grab every hour as per above procedure for 8-hours.
Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity 1.01</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp. 430°F</td>
</tr>
<tr>
<td>Boilers steam rate 33 pph</td>
</tr>
<tr>
<td>Boilers running condition Normal: Y/N * Any important change</td>
</tr>
</tbody>
</table>

Fri. June 8

07:30-10:00 AM

Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.
Follow QA and Chain of Custody procedures.

Thank you,

Anil Mehrotra
Plant Engineer


**REPORT OF ANALYSIS**

**CLIENT:** Hampton/NASA Steam Plant  
**ATTN:** Anil Mehrotra, Plant Engineer  
**ADDRESS:** 50 Wythe Creek Road  
**CITY:** Hampton, VA 23666  
**PHONE:** (757) 865-1914  
**FAX:** e: amehrotra@hampton.gov  
**SPECIAL NOTES:**  

**SAMPLE RECEIPT**  
**DATE:** 6/10/2016 **TIME:** 1350  
**GRAB COLLECTION**  
**DATE:** 6/8/2016 **TIME:** 0000  
**COLLECTED BY:** CLIENT  
**PICK UP BY:** REED - DB  
**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION** ✓ Good  
**REPORT NO:** 16-10338 14:19  

**SAMPLE ID:** HSP-0616-SST5  
**SAMPLE NO:** 16-10338

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt;0.050</td>
<td>mg/L</td>
<td>PEJ 06/16/16 1435</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.478</td>
<td>mg/L</td>
<td>PEJ 06/16/16 1435</td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.368</td>
<td>mg/L</td>
<td>PEJ 06/16/16 1435</td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt;0.010</td>
<td>mg/L</td>
<td>PEJ 06/16/16 1435</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>0.152</td>
<td>mg/L</td>
<td>PEJ 06/16/16 1435</td>
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<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt;0.0002</td>
<td>mg/L</td>
<td>TLG 09/17/16 1125</td>
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<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.050</td>
<td>1</td>
<td>&lt;0.050</td>
<td>mg/L</td>
<td>PEJ 06/16/16 1435</td>
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<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt;0.010</td>
<td>mg/L</td>
<td>PEJ 06/16/16 1435</td>
</tr>
</tbody>
</table>

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James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498  

VELAP# 460013  
EPA# VA00015  

Page 1 of 2
REPORT OF ANALYSIS

NOTES:
JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.
Reproduction of this report is not permitted, except in full, without written approval from James R. Reed & Associates.
The results on this report relate only to the sample(s) provided for analysis.
Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.62
Extraction Fluid used: #2
Final (end point) pH of Extraction Fluid: 5.83

Authorized By: [Signature]
Elaine Claiborne, Laboratory Director
Date: 17-Jun-16
## ANALYSIS SAMPLE CHAIN OF CUSTODY

**SAMPLE NUMBER:** HSP 0616-5575  
**MATERIAL:** Residue Ash  
**SAMPLE PREPARED BY:** Martin Benfield  
**METHOD:** HSP-3A

<table>
<thead>
<tr>
<th>CUSTODIAN</th>
<th>FROM</th>
<th>TO</th>
<th>Q. A. Weight</th>
<th>Test</th>
</tr>
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<tbody>
<tr>
<td>Martin Benfield</td>
<td>0700</td>
<td>0840</td>
<td>225.48</td>
<td>TCLP</td>
</tr>
<tr>
<td></td>
<td>0840</td>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0950</td>
<td>1350</td>
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<tr>
<td></td>
<td>1350</td>
<td>03/17/16</td>
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<td>03/17/16</td>
<td>06/18/16</td>
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</table>

Reviewed By:  
Date: 06/17/16

Hampton/NASA Steam Plant  
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
### Residue ash Sample Data Records

**Sample #** HSP-06670 - 5657

**Method Used:** HSP-3-A

**Sample Certified Valid:** [Signature]

<table>
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<td>14:02</td>
<td>15:02</td>
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<td>19:00</td>
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<td>X</td>
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<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
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<td>[Signature]</td>
<td>[Signature]</td>
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<tr>
<td>Initials</td>
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</table>

### WEIGHT TALLY SHEET (+ or - ½ Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 34 ½</td>
<td>15. 35</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 47</td>
<td>16. 35</td>
<td>1. 47 ½</td>
</tr>
<tr>
<td>3. 48</td>
<td>17. 32 ½</td>
<td>2. 39 ½</td>
</tr>
<tr>
<td>4. 87 ½</td>
<td>18. 33</td>
<td>3. 33</td>
</tr>
<tr>
<td>5. 34 ½</td>
<td>19. 34 ½</td>
<td>4. 34 ½</td>
</tr>
<tr>
<td>6. 34 ½</td>
<td>20. 35</td>
<td>5.</td>
</tr>
<tr>
<td>7. 35</td>
<td>21. 35</td>
<td>6.</td>
</tr>
<tr>
<td>8. 29 ½</td>
<td>22. 35</td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>9. 30 ½</td>
<td>23. 35</td>
<td>1. 35</td>
</tr>
<tr>
<td>10. 43</td>
<td>24. 40</td>
<td>2.</td>
</tr>
<tr>
<td>11. 35</td>
<td>25. 34</td>
<td>3.</td>
</tr>
<tr>
<td>13. 40</td>
<td>27. 34</td>
<td>5.</td>
</tr>
<tr>
<td>14. 34</td>
<td>28. 34</td>
<td>6.</td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample # HSP-0666-5ST5

<table>
<thead>
<tr>
<th>Wet Trench pH: 8.7</th>
<th>Boilers Operating: #1, #2</th>
<th>Weather: Sunny, clear</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Treatment/ Injection Rate: Sodium Silicide @ 16.0 gal/day</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Comments: Reagent Spec. Grav. 1.01</td>
<td></td>
<td></td>
</tr>
<tr>
<td>FF Inlet Temp: 430°F</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Steam Rate: 33 lbs/h</td>
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<tr>
<td>FF Modules on 2, 3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SO2 Control: 60</td>
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</tbody>
</table>

Team(s): A Team

Quality Leader: Name: Jack Dye Designation: AO

Team Leader: Name: Martin Benfield Designation: OE

<table>
<thead>
<tr>
<th>Mass Proportion Samples</th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>62.1</td>
<td>79.31</td>
<td>16.0</td>
<td></td>
</tr>
<tr>
<td>Unburnt</td>
<td>144.5</td>
<td>18.45</td>
<td>3.5</td>
<td>Total: 22.5 Lbs.</td>
</tr>
<tr>
<td>Unburnable</td>
<td>17.5</td>
<td>2.23</td>
<td>0.5</td>
<td>With bucket and lid.</td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: Martin Benfield

Date: 6/8/16
Calculation Sheet

20 lb. Ash Sample

Sample #: HSP-D616 - SST5
Date: 6/8/16

<table>
<thead>
<tr>
<th>1</th>
<th>Sub Total (Lbs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Aggregate</td>
<td>62.1</td>
</tr>
<tr>
<td>b. Unburnt</td>
<td>144.5</td>
</tr>
<tr>
<td>c. Metal</td>
<td>17.5</td>
</tr>
<tr>
<td>d. Total</td>
<td>78.3</td>
</tr>
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</table>

<table>
<thead>
<tr>
<th>2</th>
<th>Mass Proportion (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Aggregate ((\frac{a}{d} \times 100%))</td>
<td>79.31</td>
</tr>
<tr>
<td>B. Unburnt ((\frac{b}{d} \times 100%))</td>
<td>18.45</td>
</tr>
<tr>
<td>C. Metal ((\frac{c}{d} \times 100%))</td>
<td>2.23</td>
</tr>
<tr>
<td>D. total</td>
<td>100%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>3</th>
<th>Calculate 20 lb Protection (Lb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate ((A \times 20\text{Lb}))</td>
<td>15.86</td>
</tr>
<tr>
<td>Unburnt ((B \times 20\text{Lb}))</td>
<td>3.69</td>
</tr>
<tr>
<td>Metal ((C \times 20\text{Lb}))</td>
<td>4.4</td>
</tr>
<tr>
<td>Total</td>
<td>20.0</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>4</th>
<th>Rounded Up/Down (Lbs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>16.0 Lb</td>
</tr>
<tr>
<td>Unburnt</td>
<td>3.5 Lb</td>
</tr>
<tr>
<td>Metal</td>
<td>1.5 Lb</td>
</tr>
<tr>
<td>Total</td>
<td>20.0 Lb</td>
</tr>
</tbody>
</table>
Hampton/NASA Steam Plant

Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0616–SST6

Sample Collection: Wed. June 8, 2016
QA Sample Preparation: Thu. June 9, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon Wed. June 8, 2016 & will continue until 19:00 hrs Thu. June 9, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP-3A according to the following schedule and Quality Assurance procedures:

Wed. June 8:

AM Team

Starting:
12:00 noon

Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition.

Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Ending:
19:00 hrs

Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Continue 1-minute residue ash grab every hour as per above procedure for 8-hours.

Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (Set these values after 7:00 pm on 6/7)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>


Thu. June 9

07:30–10:00 AM
Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample. Follow QA and Chain of Custody procedures.

Thank you,

Anil Mebrotra
Plant Engineer
REPORT OF ANALYSIS

CLIENT: Hampton/NASA Steam Plant
ATTN: Anil Mehrotra, Plant Engineer
ADDRESS: 50 Wyche Creek Road
CITY: Hampton, VA 23666
PHONE: (757) 865-1914
FAX: emehrotra@hampton.gov

SAMPLE RECEIPT
DATE: 6/1/2016 TIME: 11:50

GRAB COLLECTION
DATE: 6/9/2016 TIME: 00:00

COLLECTED BY: CLIENT
PICK UP BY: REED-DB

NUMBER OF CONTAINERS: 1
GOOD CONDITION ☑ Good ☐ Other (See C-O-C)

REPORT NO: 16-10339 14:19

SAMPLE ID: HSP-0616-SST6
SAMPLE NO 16-10339

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JQA Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Toxic Characteristic Leaching Procedure by SW-846 Method 1311</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt;0.050 mg/L</td>
<td>PEJ 06/16/16 1407</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.602 mg/L</td>
<td>PEJ 06/16/16 1407</td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.137 mg/L</td>
<td>PEJ 06/16/16 1407</td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt;0.010 mg/L</td>
<td>PEJ 06/16/16 1407</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt;0.050 mg/L</td>
<td>PEJ 06/16/16 1407</td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt;0.0002 mg/L</td>
<td>TLG 06/17/16 1110</td>
</tr>
<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.010</td>
<td>1</td>
<td>&lt;0.050 mg/L</td>
<td>PEJ 06/16/16 1407</td>
</tr>
<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt;0.010 mg/L</td>
<td>PEJ 06/16/16 1407</td>
</tr>
</tbody>
</table>

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax (757) 873-1498

VELAP# 460013
EPA# VA00015

Page 1 of 2
REPORT OF ANALYSIS

NOTES:
JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.
Reproduction of this report is not permitted, except in full, without written approval from James R. Reed & Associates.
The results on this report relate only to the sample(s) provided for analysis.
Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 10.97
Extraction Fluid used: #1
Final (end point) pH of Extraction Fluid: 7.35

Authorized By: [Signature]
Elaine Claiborne, Laboratory Director
Date: 17-Jun-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax: (757) 873-1498

VELAP# 460013
EPA# VA00015

Page 2 of 2
<table>
<thead>
<tr>
<th>Custodian</th>
<th>From</th>
<th>To</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jack Dye</td>
<td>0700</td>
<td>9 Jun 16</td>
<td>HSP-3A</td>
</tr>
<tr>
<td>Bill Leonard</td>
<td>1005</td>
<td>9 Jun 16</td>
<td>0950</td>
</tr>
<tr>
<td>Jack Dye</td>
<td>0950</td>
<td>6-10-16</td>
<td>1350</td>
</tr>
<tr>
<td>Jack Dye</td>
<td>6-10-16</td>
<td>01/350</td>
<td></td>
</tr>
</tbody>
</table>

Q. A. Weight: 22.5 lbs
Test: [Results]

Reviewed By: [Signature]
Date: 6/17/16

Hampton/NASA Steam Plant
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

<table>
<thead>
<tr>
<th>SAMPLE #</th>
<th>Sample Date:</th>
<th>Method Used:</th>
</tr>
</thead>
<tbody>
<tr>
<td>HSP-06/14</td>
<td>7 Jun 78</td>
<td>HSP-3A</td>
</tr>
</tbody>
</table>

Sample Certified Valid:

<table>
<thead>
<tr>
<th>GRAB</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
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<tbody>
<tr>
<td>Time</td>
<td>120</td>
<td>131</td>
<td>140</td>
<td>150</td>
<td>140</td>
<td>170</td>
<td>180</td>
<td>191</td>
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<tr>
<td>Initials</td>
<td>17</td>
<td>18</td>
<td>19</td>
<td>20</td>
<td>21</td>
<td>22</td>
<td>23</td>
<td>24</td>
</tr>
</tbody>
</table>

### WEIGHT TALLY SHEET (+ or - 1/2 Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
<th>Unburnt Combustibles</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 35</td>
<td>15. 39.5</td>
<td>1. 35.5</td>
<td>2. 39</td>
</tr>
<tr>
<td>2. 39</td>
<td>16. 43.5</td>
<td>2. 39.5</td>
<td>3. 39.5</td>
</tr>
<tr>
<td>3. 37.5</td>
<td>17. 35.5</td>
<td>3. 37.5</td>
<td>4. 37.5</td>
</tr>
<tr>
<td>4. 41</td>
<td>18. 28.5</td>
<td>4. 27</td>
<td></td>
</tr>
<tr>
<td>5. 44.5</td>
<td>19. 33.5</td>
<td>5. 37.5 (Metal)</td>
<td></td>
</tr>
<tr>
<td>6. 46.5</td>
<td>20. 37</td>
<td>6. 37.5</td>
<td></td>
</tr>
<tr>
<td>7. 53</td>
<td>21. 28.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8. 54.5</td>
<td>22. 28</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9. 55</td>
<td>23. 28.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10. 55</td>
<td>24. 28</td>
<td></td>
<td></td>
</tr>
<tr>
<td>11. 49.5</td>
<td>25. 22.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>12. 49.5</td>
<td>26.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>13. 55</td>
<td>27.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>14. 45</td>
<td>28.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample # HSP-068-SS76

<table>
<thead>
<tr>
<th>Wet Trench pH</th>
<th>Boilers Operating</th>
<th>Weather</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.1</td>
<td>1+2</td>
<td>clean</td>
</tr>
</tbody>
</table>

Chemical Treatment/ Injection Rate: 18.0 Gals/hr/day

Comments:

---

Team(s): B

Quality Leader: Name: Jock D. Dye  Designation: AD

Team Leader: Name: Phil Gamble  Designation: OE

<table>
<thead>
<tr>
<th>Mass Proportion Samples</th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>9.56</td>
<td>71.4%</td>
<td>14.5</td>
<td>Total: 22.5 lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>3.56</td>
<td>26.58</td>
<td>5</td>
<td>With bucket and lid.</td>
</tr>
<tr>
<td>Unburnable</td>
<td>2.65</td>
<td>1.97</td>
<td>0.5</td>
<td></td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: [Signature]
Date: 6/9/10
**Calculation Sheet**

**20 lb. Ash Sample**

Sample #: 415 P 06/16-557 B

Date: 6/9/16

---

### Mass Proportion (%)

- **a. Aggregate** ($\frac{a}{a}$ x 100%) = 71.43%
- **b. Unburnt** ($\frac{b}{b}$ x 100%) = 26.58%
- **c. Metal** ($\frac{c}{c}$ x 100%) = 1.97%
- **d. Total** = 100%

---

### Sub Total (lbs)

- **a. Aggregate** 956.5 lbs
- **b. Unburnt** 356.0 lbs
- **c. Metal** 26.5 lbs
- **d. Total** 1339.0 lbs

---

### Calculate 20 lb Proportion (lbs)

- **Aggregate** ($A \times 20\text{ lb}$) = 14.5 lb
- **Unburnt** ($B \times 20\text{ lb}$) = 5.3 lb
- **Metal** ($C \times 20\text{ lb}$) = 0.39 lb
- **Total** = 20 lb

---

### Rounded Up/down (lbs)

- **Aggregate** 14.5 lb
- **Unburnt** 5.0 lb
- **Metal** 2.5 lb
- **Total** 20.0 lb
Hampton/NASA Steam Plant

Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP-0616-SST7

Sample Collection: Thu. June 9, 2016
QA Sample Preparation: Fri. June 10, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon Thu. June 9, 2016 & will continue until 19:00 hrs Thu. June 9, 2016.
The sample will be gathered, prepared, and sealed for analysis as per Method HSP-3A according to the following schedule and Quality Assurance procedures:

Thu. June 9:

AM Team

Starting:
12:00 noon

Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition.
Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Ending:
19:00 hrs

Continue 1-minute residue ash grab every hour as per above procedure for 8-hours.
Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

| Test Day Control Parameters (Set these values after 7:00 pm on 6/8) |
|-------------------------------------------------|-----------------|-----------------|
| Reagent Specific Gravity                       | 1.01            | SO₂ Control     | 60 ppm          |
| Fabric Filter Inlet Temp.                      | 430°F           | FF Modules on   | Mod 1, 3        |
| Boilers steam rate                             | 33 pph          | Sodium Sulfide rate | 20.0 g/day |
| Boilers running condition                      | Normal: Y/N    | * Any important change |

Remarks:
* 1. Mechanical issues
  2. Upset condition: trench cleaning/ wet trash
  3. Ash conveyor issues
  4. Others:

Fri. June 10
07:30–10:00 AM Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.
Follow QA and Chain of Custody procedures.

Thank you,

Anil Mehrotra
Plant Engineer

After last sample collected place in FF modules in service.
### REPORT OF ANALYSIS

**CLIENT:** Hampton/NASA Steam Plant  
**ATTN:** Anil Mehrotra, Plant Engineer  
**ADDRESS:** 50 Wythe Creek Road  
**CITY:** Hampton, VA 23666  
**PHONE:** (757) 865-1914  
**FAX:** e: amehrotra@hampton.gov  

**SAMPLE RECEIPT**  
**DATE:** 6/10/2016  
**TIME:** 1350  
**GRAB COLLECTION**  
**DATE:** 6/10/2016  
**TIME:** 0000  
**COLLECTED BY:** CLIENT  
**PICK UP BY:** REED - DB  
**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION:** ☑️ Good  
**Other (See C-O-C):**  
**REPORT NO:** 16-10340 14:19

---

**SAMPLE ID:** HSP-0616-SST7  
**SAMPLE NO:** 16-10340

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analysis/Date/Time</th>
</tr>
</thead>
</table>
| Toxic Characteristic Leaching Procedure by SW-846 Method 1311  
Arsenic            | D004       | 6010C         | 0.050  | 5                | <0.050 | mg/L  | PEJ 06/16/16 1429 |
| Barium             | D005       | 6010C         | 0.250  | 100              | 0.364  | mg/L  | PEJ 06/16/16 1429 |
| Cadmium            | D006       | 6010C         | 0.005  | 1                | <0.005 | mg/L  | PEJ 06/16/16 1429 |
| Chromium           | D007       | 6010C         | 0.010  | 5                | <0.010 | mg/L  | PEJ 06/16/16 1429 |
| Lead               | D008       | 6010C         | 0.050  | 5                | <0.050 | mg/L  | PEJ 06/16/16 1429 |
| Mercury            | D009       | 7470A         | 0.0002 | 0.2              | <0.0002| mg/L  | TLG 06/17/16 1113 |
| Selenium           | D010       | 6010C         | 0.050  | 1                | <0.050 | mg/L  | PEJ 06/16/16 1429 |
| Silver             | D011       | 6010C         | 0.010  | 5                | <0.010 | mg/L  | PEJ 06/16/16 1429 |

---

James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498  

VELAP# 460013  
EPA# VA00015

Page 1 of 2
REPORT OF ANALYSIS

NOTES:
JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.

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The results on this report relate only to the sample(s) provided for analysis.

Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.18
Extraction Fluid used: #1
Final (end point) pH of Extraction Fluid: 8.01

Authorized By: [Signature]
Elaine Claiborne, Laboratory Director
Date: 17-Jun-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4705 • Fax: (757) 873-1498

VELAP# 460013
EPA# VA00015

Page 2 of 2
ANALYSIS SAMPLE
CHAIN OF CUSTODY

SAMPLE NUMBER: HSP-0616-55T7
MATERIAL: Residue, Ash

SAMPLE PREPARED BY: Jack Dye
METHOD: HSP-3A

<table>
<thead>
<tr>
<th>CUSTODIAN</th>
<th>FROM</th>
<th>TO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Martin Barkfield</td>
<td>0700</td>
<td>0840</td>
</tr>
<tr>
<td></td>
<td>0840</td>
<td>0950</td>
</tr>
<tr>
<td>Dona Lee Boyal</td>
<td>0950</td>
<td>1350</td>
</tr>
</tbody>
</table>

Q.A. Weight: 22.6 lbs
Test: TCLP
All Metals

Reviewed By: [Signature]
Date: 6/10/16

Hampton/NASA Steam Plant
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

**Sample #**: HSP-0616
**Sample Date**: 6/9/16
**Method Used**: HSP-3A

**Sample Certified Valid**: [Signature]

<table>
<thead>
<tr>
<th>GRAB</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
<th>13</th>
<th>14</th>
<th>15</th>
<th>16</th>
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</thead>
<tbody>
<tr>
<td>Time</td>
<td>13:25</td>
<td>13:35</td>
<td>14:02</td>
<td>14:07</td>
<td>16:12</td>
<td>17:00</td>
<td>18:11</td>
<td>19:06</td>
</tr>
<tr>
<td>Initials</td>
<td>AAB</td>
<td>BCC</td>
<td>DDD</td>
<td>EEE</td>
<td>FFF</td>
<td>GGG</td>
<td>HHH</td>
<td>IJK</td>
</tr>
</tbody>
</table>

---

### WEIGHT TALLY SHEET (+ or - 1/2 Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 38.5</td>
<td>15. 34.5</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 37</td>
<td>16. 29</td>
<td>1. 43</td>
</tr>
<tr>
<td>3. 45.5</td>
<td>17. 31</td>
<td>2. 29.5</td>
</tr>
<tr>
<td>4. 79</td>
<td>18. 32</td>
<td>3. 82</td>
</tr>
<tr>
<td>5. 37.5</td>
<td>19. 30</td>
<td>4. 24</td>
</tr>
<tr>
<td>6. 36.5</td>
<td>20. 32</td>
<td>5. 21</td>
</tr>
<tr>
<td>7. 39</td>
<td>21. 23</td>
<td>6. 12</td>
</tr>
<tr>
<td>8. 46.5</td>
<td>22.</td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>9. 38.5</td>
<td>23.</td>
<td></td>
</tr>
<tr>
<td>10. 35.5</td>
<td>24.</td>
<td>1. 23</td>
</tr>
<tr>
<td>11. 36.5</td>
<td>25.</td>
<td>2. 2</td>
</tr>
<tr>
<td>12. 38</td>
<td>26.</td>
<td>3.</td>
</tr>
<tr>
<td>13. 38.5</td>
<td>27.</td>
<td>4.</td>
</tr>
<tr>
<td>14. 38</td>
<td>28.</td>
<td>5.</td>
</tr>
</tbody>
</table>

(continued on the next page)
Residue ash Sample Data Records
(Please Sign and Date below)

Sample # 458-0016- SST 7

<table>
<thead>
<tr>
<th>Wet Trench pH</th>
<th>Boilers Operating</th>
<th>Weather</th>
<th>Chemical Treatment/Injection Rate</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.9</td>
<td>1/2</td>
<td>Clear, Sunny</td>
<td>Sodium Sulphide</td>
<td>Reagent Spec. Gravity 1.01, FF inlet temp. 430°F, Boilers Steam Rate 33 ppb, SO2 control 60 ppm, FF modules on 1, 3</td>
</tr>
</tbody>
</table>

Team(s): A and B

Quality Leader: Name Jack Dye Designation: A0

Team Leader: Name Martin Banfield Designation: OE

### Mass Proportion Samples

<table>
<thead>
<tr>
<th></th>
<th>Lbs.</th>
<th>%</th>
<th>2 C. Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>744</td>
<td>80.1</td>
<td>16</td>
<td>Total: 12.5 Lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>162</td>
<td>17.4</td>
<td>3.5</td>
<td>With bucket and lid.</td>
</tr>
<tr>
<td>Unburnable</td>
<td>23</td>
<td>2.5</td>
<td>0.5</td>
<td></td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: Martin Banfield

Date: 07/10/16
Hampton/NASA Steam Plant

Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0616–SST8

Sample Collection: Fri. June 10, 2016
QA Sample Preparation: Mon. June 13, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon Fri. June 10, 2016 & will continue until 19:00 hrs Fri. June 10, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to the following schedule and Quality Assurance procedures:

Fri. June 10:
AM Team
Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Starting:
12:00 noon → Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Ending:
19:00 hrs
Continue 1-minute residue ash grab every hour as per above procedure for 8-hours. Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

| Test Day Control Parameters (Set these values after 7:00 pm on 6/9) |
|---|---|---|
| Reagent Specific Gravity | 1.01 | SO₂ Control | 60 ppm |
| Fabric Filter Inlet Temp. | 400°F | FF Modules on | Mod 2, 3 |
| Boilers steam rate | 33 ppm | Sodium Sulfide rate | 24.0 g/day |
| Boilers running condition | Normal: Y/N * | Any important change | |


Mon. June 13
07:30–10:00 AM Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample. Follow QA and Chain of Custody procedures.

Thank you,
Anil Mehrotra
Plant Engineer

When last sample collected resume operation with all modules in the fabric filters
# REPORT OF ANALYSIS

**CLIENT:** Hampton/NASA Steam Plant  
**SAMPLE RECEIPT**

**ATTN:** Anil Mehrotra, Plant Engineer  
**DATE:** 6/15/2016  
**TIME:** 12:15

**ADDRESS:** 50 Wythe Creek Road  
**GRAB COLLECTION**

**CITY:** Hampton, VA 23666  
**DATE:** 6/13/2016  
**TIME:** 00:00

**PHONE:** (757) 865-1914  
**COLLECTED BY:** CLIENT

**FAX:** e: amehrotra@hampton.gov  
**PICK UP BY:** REED - TL

**SPECIAL NOTES:**  
**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION** ☑ Good ☐ Other (See C-O-C)  
**REPORT NO:** 16-10592 14:19

---

**SAMPLE ID:** HSP-0616-SST8  
**SAMPLE NO:** 16-10592

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1110</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>&lt; 0.250</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1110</td>
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<tr>
<td>Cadmium</td>
<td>D006</td>
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<td>0.065</td>
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<td>PEJ 06/17/16 1110</td>
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<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
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<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1110</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
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<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1110</td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt; 0.0002</td>
<td>mg/L</td>
<td>TLG 06/17/16 1148</td>
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<td>Selenium</td>
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<td>6010C</td>
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<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1110</td>
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<tr>
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<td>6010C</td>
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<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1110</td>
</tr>
</tbody>
</table>

---

**James R. Reed & Associates**  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498

**VELAP# 460013**  
**EPA# VA00015**
REPORT OF ANALYSIS

NOTES:

JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.

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The results on this report relate only to the sample(s) provided for analysis.

Results conform to NELAC standards, when applicable, unless otherwise indicated.

Initial pH: 11.08
Extraction Fluid used: #1
Final (end point) pH of Extraction Fluid: 7.30

Authorized By: [Signature]
Elaine Claiborne, Laboratory Director
Date: 17-Jun-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax: (757) 873-1498

VELAP# 460013
EPA# VA00015

Page 2 of 2
## ANALYSIS SAMPLE CHAIN OF CUSTODY

**Sample Number:** H5P-06-14-5578  
**Material:**  
**Sample Prepared By:** Donald Wooladon  
**Method:** H5P-3A

<table>
<thead>
<tr>
<th>Custodian</th>
<th>From</th>
<th>To</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>Donald Wooladon</td>
<td>3/16/16</td>
<td>07/01</td>
<td>03/16/16</td>
</tr>
<tr>
<td></td>
<td>6/13/16</td>
<td>09/50</td>
<td>15/06/16</td>
</tr>
<tr>
<td>TSA-00</td>
<td>6/15/16</td>
<td>11/50</td>
<td>6/15/16</td>
</tr>
</tbody>
</table>

Q. A. Weight: 22.5 lbs  
Reviewed By: [Signature]  
Date: 6/13/16

---

Hampton/NASA Steam Plant  
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
# Hampton/NASA Steam Plant
Residue ash Sample Data Records

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Sample Certified Valid</th>
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<tbody>
<tr>
<td>HSP-014</td>
<td>10 June 16</td>
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<table>
<thead>
<tr>
<th>Sample Date:</th>
<th>Method Used:</th>
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<tbody>
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<td>10 June 16</td>
<td>HSP-3A</td>
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<td>13:00</td>
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<td>16:00</td>
<td>17:00</td>
<td>18:15</td>
<td>19:10</td>
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<td>N</td>
<td>X</td>
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<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
<th>13</th>
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<th>16</th>
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</thead>
<tbody>
<tr>
<td>Time</td>
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<td>Initials</td>
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<table>
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<th>21</th>
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<tr>
<td>Time</td>
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<td></td>
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<td></td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>

## WEIGHT TALLY SHEET (+ or - ½ Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>70.5 lbs</td>
<td>15.</td>
</tr>
<tr>
<td>2.</td>
<td>43.5 lbs</td>
<td>16.</td>
</tr>
<tr>
<td>3.</td>
<td>46 lbs</td>
<td>17.</td>
</tr>
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<td>4.</td>
<td>48 lbs</td>
<td>18.</td>
</tr>
<tr>
<td>5.</td>
<td>49.5 lbs</td>
<td>19.</td>
</tr>
<tr>
<td>6.</td>
<td>54 lbs</td>
<td>20.</td>
</tr>
<tr>
<td>7.</td>
<td>47.5 lbs</td>
<td>21.</td>
</tr>
<tr>
<td>8.</td>
<td>58 lbs</td>
<td>22.</td>
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<tr>
<td>9.</td>
<td>55 lbs</td>
<td>23.</td>
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<tr>
<td>10.</td>
<td>42.5 lbs</td>
<td>24.</td>
</tr>
<tr>
<td>11.</td>
<td>41.5 lbs</td>
<td>25.</td>
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<tr>
<td>12.</td>
<td>44.5 lbs</td>
<td>26.</td>
</tr>
<tr>
<td>13.</td>
<td>35 lbs</td>
<td>27.</td>
</tr>
<tr>
<td>14.</td>
<td>27.5 lbs</td>
<td>28.</td>
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</table>

<table>
<thead>
<tr>
<th>Other Materials</th>
<th>Unburnt Combustibles</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>33 lbs</td>
</tr>
<tr>
<td>2.</td>
<td>32 lbs</td>
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<table>
<thead>
<tr>
<th>Other Materials</th>
<th>Unburnable (Metal)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>21 lbs</td>
</tr>
<tr>
<td>2.</td>
<td></td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample # 155-0416-5576

Wet Trench pH: 9.1  Boilers Operating: 1+2  Weather: Cloudy

Chemical Treatment/Injection Rate: Sodium Sulphate TDS 440 gpd

Comments:

Team(s): A. Smith, R Darrah

Quality Leader: Name David Date Designation: Ash Handler

Team Leader: Name Donald Weevers Designation: OP Eng

Mass Proportion Samples

<table>
<thead>
<tr>
<th></th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>463</td>
<td>88.5</td>
<td>175 / 165</td>
<td>Total: 22.5 Lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>65</td>
<td>8.7</td>
<td>2.0 / 165</td>
<td>With bucket and lid.</td>
</tr>
<tr>
<td>Unburnable</td>
<td>21</td>
<td>2.8</td>
<td>0.5 / 165</td>
<td></td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: [Signature]
Date: 6/11/11
### Calculation Sheet

**20 lb. Ash Sample**

<table>
<thead>
<tr>
<th>Sample #</th>
<th>H5P-0616-SST8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Date</td>
<td>6/12/16</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sub Total (Lbs)</th>
<th>Mass Proportion (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Aggregate</td>
<td>6.63</td>
</tr>
<tr>
<td>b. Unburnt</td>
<td>6.5</td>
</tr>
<tr>
<td>c. Metal</td>
<td>2.1</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>74.9</strong></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>(3) Calculate 20 lb Proportion (Lb)</th>
<th>(4) Rounded Up/Down (Lb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate ($\frac{a}{74.9} \times 20$)</td>
<td>17.70</td>
</tr>
<tr>
<td>Unburnt ($\frac{b}{74.9} \times 20$)</td>
<td>17.30</td>
</tr>
<tr>
<td>Metal ($\frac{c}{74.9} \times 20$)</td>
<td>5.80</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>20 lb</strong></td>
</tr>
</tbody>
</table>

| Aggregate | 17.50 lb |
| Unburnt   | 2.00 lb  |
| Metal     | 5.00 lb  |
| **Total** | **20.00 lb** |
Hampton/NASA Steam Plant

Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0616–SST9

Sample Collection: Mon. June 13, 2016
QA Sample Preparation: Tue. June 14, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon Mon. June 13, 2016 & will continue until 19:00 hrs Mon. June 13, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to the following schedule and Quality Assurance procedures:

Mon. June 13:
AM Team
Starting:
12:00 noon
Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Ending:
19:00 hrs
Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Continue 1-minute residue ash grab every hour as per above procedure for 8-hours.
Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (After last sample collection on 6/10)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>


Tue. June 14
07:30–10:00 AM Use Method HSP–3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample. Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.

Thank you,

Anil Mehrotra
Plant Engineer
REPORT OF ANALYSIS

CLIENT: Hampton/NASA Steam Plant
ATTN: Anil Mehrotra, Plant Engineer
ADDRESS: 50 Wythe Creek Road
CITY: Hampton, VA 23666
PHONE: (757) 865-1914
FAX: e: amehrotra@hampton.gov

SAMPLE RECEIPT
DATE: 6/15/2016 TIME: 1215
GRAB COLLECTION
DATE: 6/14/2016 TIME: 0000
COLLECTED BY: CLIENT
PICK UP BY: REED - TL
NUMBER OF CONTAINERS: 1
GOOD CONDITION ☑ Good ☐ Other (See C-O-C)
REPORT NO: 16-10593 14:19

SAMPLE ID: HSP-0616-SST9
SAMPLE NO 16-10593

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analysis/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1202</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.704 M</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1202</td>
</tr>
<tr>
<td>Cadmium</td>
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<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.373 M</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1202</td>
</tr>
<tr>
<td>Chromium</td>
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<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>0.173</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1202</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>1.35 M</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1202</td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>0.0006</td>
<td>mg/L</td>
<td>TLG 06/17/16 1223</td>
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<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.050</td>
<td>1</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1202</td>
</tr>
<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/17/16 1202</td>
</tr>
</tbody>
</table>

---

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax: (757) 873-1498
VELAP# 460013
EPA# VA00015
Page 1 of 2
REPORT OF ANALYSIS

NOTES:
JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.
Reproduction of this report is not permitted, except in full, without written approval from James R. Reed & Associates.
The results on this report refer only to the sample(s) provided for analysis.
Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.96
Extraction Fluid used: #2
Final (end point) pH of Extraction Fluid: 5.44
M Matrix spike % recovery outside acceptance range.

Authorized By: [Signature]
Elaine Claiborne, Laboratory Director
Date: 17-Jun-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax: (757) 873-1498

Page 2 of 2
# ANALYSIS SAMPLE
## CHAIN OF CUSTODY

**SAMPLE NUMBER:** HSP-016-5579  
**MATERIAL:** Ash residue  
**SAMPLE PREPARED BY:** C-D  
**METHOD:** HSP-3A

<table>
<thead>
<tr>
<th>CUSTODIAN</th>
<th>FRCM</th>
<th>TO</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>6/14/14</td>
<td>0850</td>
</tr>
<tr>
<td></td>
<td>6/14/14</td>
<td>0853</td>
</tr>
<tr>
<td>Tina Green</td>
<td>6/15/16</td>
<td>1150</td>
</tr>
</tbody>
</table>

Q.A. Weight: 225  
Test: TCLP, All metals

Reviewed By:  
Date: 6/20/16

---

Hampton/NASA Steam Plant  
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
<table>
<thead>
<tr>
<th>SAMPLE #</th>
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<th>Sampling Date: 6/14/14</th>
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<table>
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<tbody>
<tr>
<td>Time</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initials</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**WEIGHT TALLY SHEET (+ or - 1/2 Lb)**

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 37.5</td>
<td>15.</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 39</td>
<td>16.</td>
<td>1. 17</td>
</tr>
<tr>
<td>3. 41.5</td>
<td>17.</td>
<td>2. 48.5</td>
</tr>
<tr>
<td>4. 52.5</td>
<td>18.</td>
<td>3.</td>
</tr>
<tr>
<td>5. 59</td>
<td>19.</td>
<td>4.</td>
</tr>
<tr>
<td>6. 60</td>
<td>20.</td>
<td>5.</td>
</tr>
<tr>
<td>7. 57</td>
<td>21.</td>
<td>6.</td>
</tr>
<tr>
<td>8. 62</td>
<td>22.</td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>9. 50.5</td>
<td>23.</td>
<td>1. 21.5</td>
</tr>
<tr>
<td>10. 52.5</td>
<td>24.</td>
<td>2.</td>
</tr>
<tr>
<td>11. 40</td>
<td>25.</td>
<td>3.</td>
</tr>
<tr>
<td>12. 48</td>
<td>26.</td>
<td>4.</td>
</tr>
<tr>
<td>13.</td>
<td>27.</td>
<td>5.</td>
</tr>
<tr>
<td>14.</td>
<td>28.</td>
<td>6.</td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample #: HSP-0416-3579

Wet Trench pH: 8.4 | Boilers Operating: 1-2 | Weather: Sunny

Chemical Treatment/ Injection Rate: 24.0 gal/day

Comments:
- Reagent Specific Gravity 1.01, SO₂ Concentration 60 ppm
- Fabric Filter Inlet Temp 400°F, F.F. Modules Mod 2.3
- Boilers steam rate - 33,000 lb/hr, Sodium Sulfide rate 4.19 gal/day
- Boilers Running Condition: Normal

Team (s):
C-D

Quality Leader: Name Taylor Designation: Ash Pad

Team Leader: Name Krajcir Designation: Ash Pad

Mass Proportion Samples

<table>
<thead>
<tr>
<th></th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>594.6</td>
<td>87.23</td>
<td>17.5</td>
<td>Total: 325 Lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>66.5</td>
<td>9.14</td>
<td>2.0</td>
<td>With bucket and lid.</td>
</tr>
<tr>
<td>Unburnable</td>
<td>21.5</td>
<td>3.15</td>
<td>1.5</td>
<td></td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: [Signature]

Date: 6/14/16
Calculation Sheet

20 lb. Ash Sample

Sample #: HSP-0616-54T9

Date: 6-14-2016

<table>
<thead>
<tr>
<th>(1) Sub Total (lbs)</th>
<th>(2) Mass Proportion (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Aggregate</td>
<td>594.5</td>
</tr>
<tr>
<td>b. Unburnt</td>
<td>65.5</td>
</tr>
<tr>
<td>c. Metal</td>
<td>21.5</td>
</tr>
<tr>
<td>d. Total</td>
<td>681.5</td>
</tr>
<tr>
<td></td>
<td>a. Aggregate (\frac{a}{d} \times 100%)</td>
</tr>
<tr>
<td></td>
<td>b. Unburnt (\frac{b}{d} \times 100%)</td>
</tr>
<tr>
<td></td>
<td>c. Metal (\frac{c}{d} \times 100%)</td>
</tr>
<tr>
<td></td>
<td>d. Total</td>
</tr>
</tbody>
</table>

(3) Calculate 20 lb Proportion (lb)

\[ \text{Aggregate} (A \times 20 \text{ lb}) \quad 17.5 \quad \text{lb.} \]
\[ \text{Unburnt} (B \times 20 \text{ lb}) \quad 1.92 \quad \text{lb} \]
\[ \text{Metal} (C \times 20 \text{ lb}) \quad 0.15 \quad \text{lb} \]
\[ \text{Total} \quad 20 \text{ lb} \]

(4) Rounded Up/Down (lbs)

\[ \text{Aggregate} \quad 17.5 \quad \text{lb.} \]
\[ \text{Unburnt} \quad 2.0 \quad \text{lb} \]
\[ \text{Metal} \quad 0.5 \quad \text{lb} \]
\[ \text{Total} \quad 20.0 \quad \text{lb} \]
Hampton/NASA Steam Plant  
Procedure for Characterization of Residue Ash treatment with Sodium Sulfide  

Sample Number: HNSP-0616-SST10  

Sample Collection: Mon. June 20, 2016  
QA Sample Preparation: Tue. June 21, 2016  

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon Mon. June 20, 2016 & will continue until 19:00 hrs Mon. June 20, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP-3A according to the following schedule and Quality Assurance procedures:

**Mon. June 20:**

**AM Team**

Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

**Starting:**

12:00 noon

Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

**Ending:**

19:00 hrs

Continue 1-minute residue ash grab every hour as per above procedure for 8-hours.

Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (After permanent Sod. Sulfide system setup on 6/17)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>

**Remarks:**

1. Mechanical issues
2. Upset condition: trench cleaning/wet trash
3. Ash conveyor issues
4. Others

**Tue. June 21**

07:30–10:00 AM  Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.

Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.

Thank you.

Anil Mehrotra  
Plant Engineer
<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA SW No.</th>
<th>Method Number</th>
<th>IRA Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Toxic Characteristic Leaching Procedure by SW-846 Method 1311</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1509</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>0.010</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1509</td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>0.450</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1509</td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>0.200</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1509</td>
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<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>0.040</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1509</td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.0004</td>
<td>mg/L</td>
<td>TLG 06/30/16 1642</td>
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<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.050</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1509</td>
</tr>
<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1509</td>
</tr>
</tbody>
</table>
NOTES:
JRA Quantitation Level is the concentration of the lowest calibration standard above zero with a reliable signal.

Reproduction of this report is not permitted, except in full, without written approval from James R. Reed & Associates.

The results on this report relate only to the sample(s) provided for analysis.

Results conform to NELAC standards, when applicable, unless otherwise indicated.

Initial pH: 11.54

Extracting Fluid used:

Final (end point) pH of Extraction Fluid: 5.51

84% Spike recovery is within acceptance range.

Authorized By: Elaine Claiborne, Laboratory Director
Date: 30-Jun-16
ANALYSIS SAMPLE
CHAIN OF CUSTODY

SAMPLE NUMBER: HASP-Y-16-SS76

MATERIAL: Kestone Ash

SAMPLE PREPARED BY: John Kosaric

METHOD: HASP-S8A

16-112047

<table>
<thead>
<tr>
<th>CUSTOMIAN</th>
<th>FROM</th>
<th>TO</th>
</tr>
</thead>
<tbody>
<tr>
<td>John Kosaric</td>
<td>1/10</td>
<td>6/21/16</td>
</tr>
<tr>
<td>8/5</td>
<td>6/21/16</td>
<td>1105</td>
</tr>
<tr>
<td>1105</td>
<td>6/21/16</td>
<td>1105</td>
</tr>
<tr>
<td>1105</td>
<td>6/22/16</td>
<td>1105</td>
</tr>
</tbody>
</table>

Q.A. Weight: 22.5 Lb

Test: TCLP ALL Metals

Reviewed By: [Signature]

Date: 7/11/16

Hampton/NASA Steam Plant
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

<table>
<thead>
<tr>
<th>SAMPLE #</th>
<th>HSP-084-5ST10</th>
<th>Sample Date: 6/25/76</th>
</tr>
</thead>
<tbody>
<tr>
<td>Method Used:</td>
<td>HSP-3A</td>
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</tr>
</tbody>
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Sample Certified Valid:

<table>
<thead>
<tr>
<th>GRAB</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initials</td>
<td>10</td>
<td>11</td>
<td>12</td>
<td>13</td>
<td>14</td>
<td>15</td>
<td>16</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>GRAB</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
<th>13</th>
<th>14</th>
<th>15</th>
<th>16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initials</td>
<td>17</td>
<td>18</td>
<td>19</td>
<td>20</td>
<td>21</td>
<td>22</td>
<td>23</td>
<td>24</td>
</tr>
</tbody>
</table>

### WEIGHT TALLY SHEET (+ or - 1/2 Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 44.5</td>
<td>15. 14.5</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 44.5</td>
<td>16. 44.0</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>3. 43</td>
<td>17. 12.5</td>
<td></td>
</tr>
<tr>
<td>4. 40.5</td>
<td>18. 3.</td>
<td></td>
</tr>
<tr>
<td>5. 44.5</td>
<td>19.</td>
<td></td>
</tr>
<tr>
<td>6. 43</td>
<td>20.</td>
<td></td>
</tr>
<tr>
<td>7. 40</td>
<td>21.</td>
<td></td>
</tr>
<tr>
<td>8. 44.5</td>
<td>22.</td>
<td></td>
</tr>
<tr>
<td>9. 39.0</td>
<td>23.</td>
<td></td>
</tr>
<tr>
<td>10. 41.0</td>
<td>24.</td>
<td></td>
</tr>
<tr>
<td>11. 26.0</td>
<td>25.</td>
<td></td>
</tr>
<tr>
<td>13.</td>
<td>27.</td>
<td></td>
</tr>
<tr>
<td>14.</td>
<td>28.</td>
<td></td>
</tr>
<tr>
<td>15.</td>
<td>29.</td>
<td></td>
</tr>
<tr>
<td>16.</td>
<td>30.</td>
<td></td>
</tr>
</tbody>
</table>

Unburnable (Metal)
### Residue ash Sample Data Records

(Please Sign and Date below)

**Sample #:** #5P-04-02-SS1D

<table>
<thead>
<tr>
<th>Wet Trench pH</th>
<th>Boilers Operating</th>
<th>Weather</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.9</td>
<td>12</td>
<td>Sunny/Clear</td>
</tr>
</tbody>
</table>

**Chemical Treatment/Injection Rate:** 13.0 g/day

**Comments:**
- #7 boiler was down + set up at 14:00
- Reagent Specific Gravity 1.01 / 50°C Control 0.00 g/m³
- F. P. Inlet Temp 140°F / F. M. Modules on Al/5
- Boiler Slm Rate 33 gph / Sodium Sulfide rate 30 g/day
- Boiler rumors condition N. Any important changes
- #2 boiler was down + back on line

**Team(s):** D Team

**Quality Leader: Name** Taylor Krajcer  
**Designation:** Ash Pad

**Team Leader: Name** Dye  
**Designation:** Ash Pad

#### Mass Proportion Samples

<table>
<thead>
<tr>
<th></th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>451.5</td>
<td>96.9</td>
<td>17.5</td>
<td>Total: 88.5 Lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>51.6</td>
<td>10.6</td>
<td>6.5</td>
<td>With bucket and lid.</td>
</tr>
<tr>
<td>Unburnable</td>
<td>11.0</td>
<td>2.1</td>
<td>2.0</td>
<td></td>
</tr>
</tbody>
</table>

**Verification by Quality Leader or Team Leader**

Sign: [Signature]  
Date: June 21, 2011
**Calculation Sheet**

**20 lb. ash sample**

Sample #: **HNSP-0616-5JT10**

Date: **6/21/2010**

<table>
<thead>
<tr>
<th></th>
<th></th>
<th><strong>Subtotal (lbs)</strong></th>
<th><strong>Mass Proportion (%)</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>a. Aggregate</td>
<td>451.5</td>
<td>A. Aggregate ((\frac{a}{d} \times 100%))</td>
<td>85.9%</td>
</tr>
<tr>
<td>b. Unburnt</td>
<td>56.5</td>
<td>B. Unburnt ((\frac{b}{d} \times 100%))</td>
<td>10.8%</td>
</tr>
<tr>
<td>c. Metal</td>
<td>11.0</td>
<td>C. Metal ((\frac{c}{d} \times 100%))</td>
<td>1.1%</td>
</tr>
<tr>
<td>d. Total</td>
<td>519.0</td>
<td>D. Total</td>
<td>100%</td>
</tr>
</tbody>
</table>

(2) Calculate 20 lb proportion (lb) (up to 2 decimal places)

<table>
<thead>
<tr>
<th></th>
<th></th>
<th><strong>Aggregate (A x 20 lb)</strong></th>
<th><strong>Unburnt (B x 20 lb)</strong></th>
<th><strong>Metal (C x 20 lb)</strong></th>
<th><strong>Total</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>7.38</td>
<td>2.14</td>
<td>1.22</td>
<td>20 lb</td>
</tr>
</tbody>
</table>

(3) Rounded up/down (lbs (to 1/2 lb))

<table>
<thead>
<tr>
<th></th>
<th></th>
<th><strong>Aggregate</strong></th>
<th><strong>Unburnt</strong></th>
<th><strong>Metal</strong></th>
<th><strong>Total</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>7.5 lb</td>
<td>2.0 lb</td>
<td>0.5 lb</td>
<td>20.0 lb</td>
</tr>
</tbody>
</table>
Hampton/NASA Steam Plant
Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP-0616-SST11

Sample Collection: Tue. June 21, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon Tue. June 21, 2016 & will continue until 19:00 hrs Tue. June 21, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP-3A according to the following schedule and Quality Assurance procedures:

Tue. June 21:
AM Team
Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Starting:
12:00 noon
Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Ending:
19:00 hrs
Continue 1-minute residue ash grab every hour as per above procedure for 8-hours.
Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (After last sample is collected on 6/20)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>


Wed. June 22
07:30–10:00 AM   Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample. Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.
Thank you,

Anil Mehrotra
Plant Engineer
<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method No.</th>
<th>QL</th>
<th>Regulatory Leve</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.05</td>
<td>&lt; 0.05</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1604</td>
<td></td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.25</td>
<td>50</td>
<td>0.431 M</td>
<td>PEJ 06/30/16 1604</td>
<td></td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.732 mg/L</td>
<td>PEJ 06/30/16 1604</td>
<td></td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>0.112</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1604</td>
</tr>
<tr>
<td>Copper</td>
<td>D008</td>
<td>6010C</td>
<td>0.05</td>
<td>3</td>
<td>9.69 mg/L</td>
<td>PEJ 06/30/16 1604</td>
<td></td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt; 0.0002</td>
<td>mg/L</td>
<td>TLG 06/30/16 1639</td>
</tr>
<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.05</td>
<td>1</td>
<td>&lt; 0.05</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1604</td>
</tr>
<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1604</td>
</tr>
</tbody>
</table>
NOTES

JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.

Reproduction of this report is not permitted, except in full, without written approval from James R. Reed & Associates.

The results on this report relate only to the sample(s) provided for analysis.

Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pllt 11.43

Extraction Field: unknown

Final (and repet.) pllt of Extraction Field: 5.36

*NOTE: spike % recovery is outside acceptance range.

Authorized By: [Signature]

Elaine Ciaburri, Laboratory Director

Date: 30-Jun-18
**ANALYSIS SAMPLE CHAIN OF CUSTODY**

**SAMPLE NUMBER:** HNSP-0416 - 05714

**SAMPLE PREPARED BY:** S. Kralcic

**METHOD:** HSP-3A

<table>
<thead>
<tr>
<th>Custodian</th>
<th>From</th>
<th>To</th>
<th>Date</th>
<th>Test</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0700</td>
<td>22 JUN 16</td>
<td>0850</td>
<td>22 JUN 16</td>
</tr>
<tr>
<td></td>
<td>0850</td>
<td>12 JUN 16</td>
<td>1105</td>
<td>22 JUN 16</td>
</tr>
<tr>
<td></td>
<td>1105</td>
<td>22 JUN 16</td>
<td>0716</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1135</td>
<td>22 JUN 16</td>
<td>0716</td>
<td></td>
</tr>
</tbody>
</table>

**Q.A. Weight:** 22.5 lb

**Test:**

---

Hampton/NASA Steam Plant
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant Residue ash Sample Data Records

**Sample #** HNSP-016-55TH  
**Method Used:** HSP-5A  
**Sample Date:** 6/22/16  
**Sample Certified Valid:**

<table>
<thead>
<tr>
<th>GRAB</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>1615</td>
<td>1705</td>
<td>1810</td>
<td>1930</td>
<td>2020</td>
<td>2125</td>
<td>2215</td>
<td>2320</td>
</tr>
<tr>
<td>Initials</td>
<td>MB</td>
<td>MB</td>
<td>MB</td>
<td>MB</td>
<td>MB</td>
<td>MB</td>
<td>MB</td>
<td>MB</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>GRAB</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
<th>13</th>
<th>14</th>
<th>15</th>
<th>16</th>
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</thead>
<tbody>
<tr>
<td>Time</td>
<td></td>
<td></td>
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</tbody>
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<table>
<thead>
<tr>
<th>GRAB</th>
<th>17</th>
<th>18</th>
<th>19</th>
<th>20</th>
<th>21</th>
<th>22</th>
<th>23</th>
<th>24</th>
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</thead>
<tbody>
<tr>
<td>Time</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td>Initials</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### WEIGHT TALLY SHEET (+ or - 1/2 Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 33.3</td>
<td>15.</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 35.6</td>
<td>16.</td>
<td>1. 43.5</td>
</tr>
<tr>
<td>3. 37.5</td>
<td>17.</td>
<td>2. 35.5</td>
</tr>
<tr>
<td>4. 34.1</td>
<td>18.</td>
<td>3. 5</td>
</tr>
<tr>
<td>5. 32.5</td>
<td>19.</td>
<td>4. 1</td>
</tr>
<tr>
<td>6. 40.2</td>
<td>20.</td>
<td>5. 2</td>
</tr>
<tr>
<td>7. 37.5</td>
<td>21.</td>
<td>e.</td>
</tr>
<tr>
<td>8. 39.3</td>
<td>22.</td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>9. 37.5</td>
<td>23.</td>
<td>1. 24.5</td>
</tr>
<tr>
<td>10. 42.4</td>
<td>24.</td>
<td>2. 1</td>
</tr>
<tr>
<td>11. 46.5</td>
<td>25.</td>
<td>3. 1</td>
</tr>
<tr>
<td>12. 41.1</td>
<td>26.</td>
<td>4. 1</td>
</tr>
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<td>13. 28.7</td>
<td>27.</td>
<td>5. 1</td>
</tr>
<tr>
<td>14. 29.5</td>
<td>28.</td>
<td>6. 1</td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample #: HUSP-0611-SST II

<table>
<thead>
<tr>
<th>Wet Trench pH: 8.6</th>
<th>Boilers Operating: 1 &amp; 2</th>
<th>Weather: clear</th>
</tr>
</thead>
</table>

Chemical Treatment/ Injection Rate: Sodium Sulphate Injection Adjusted all other parameters normal

Comments: Both boilers shutting down due to power outage, then #2 boiler restarted for during ash sample collection on 6/27/16

#1 boiler shutting down last 4 hours of sample on 6/27/16

Team(s): J. Kruza, J. Jones, D. Sopp, W. Zedernik

Quality Leader: Name John Kruza Designation: 570+II

Team Leader: Name Jackie Dye Designation: Aly Handler

<table>
<thead>
<tr>
<th>Mass Proportion Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
<tr>
<td>Lbs.</td>
</tr>
<tr>
<td>------</td>
</tr>
<tr>
<td>Aggregate</td>
</tr>
<tr>
<td>Unburnt</td>
</tr>
<tr>
<td>Unburnable</td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: [Signature]

Date: 6/27/16
**Calculation Sheet**

20 lb. Ash Sample

Sample #: HNSP-0616-SST II

Date: 6/22/2010

---

<table>
<thead>
<tr>
<th>1</th>
<th>Sub Total (lbs)</th>
<th>2</th>
<th>Mass Proportion (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Aggregate</td>
<td>512.5</td>
<td>A. Aggregate ($\frac{A}{D} \times 100%$)</td>
<td>83.19</td>
</tr>
<tr>
<td>b. Unburnt</td>
<td>79</td>
<td>B. Unburnt ($\frac{B}{D} \times 100%$)</td>
<td>12.82</td>
</tr>
<tr>
<td>c. Metal</td>
<td>24.5</td>
<td>C. Metal ($\frac{C}{D} \times 100%$)</td>
<td>3.97</td>
</tr>
<tr>
<td>d. TOTAL</td>
<td>516.0</td>
<td>D. Total</td>
<td>100%</td>
</tr>
</tbody>
</table>

---

<table>
<thead>
<tr>
<th>3</th>
<th>Calculate 20 lb Proportion (lbs) (upto 2 decimal places)</th>
<th>4</th>
<th>Rounded Up/Down (lbs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate ($A \times 20lb$)</td>
<td>16.3</td>
<td>Aggregate</td>
<td>16.5</td>
</tr>
<tr>
<td>Unburnt ($B \times 20lb$)</td>
<td>2.5</td>
<td>Unburnt</td>
<td>2.5</td>
</tr>
<tr>
<td>Metal ($C \times 20lb$)</td>
<td>7.9</td>
<td>Metal</td>
<td>1.0</td>
</tr>
<tr>
<td>Total</td>
<td>20 lb</td>
<td>Total</td>
<td>20.0 lb</td>
</tr>
</tbody>
</table>
Hampton/NASA Steam Plant
Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0616–SST12

QA Sample Preparation: Tue. June 28, 2016

An 8-hour **residue ash sample collection** with Sodium Sulfide injection is to start after boiler start-up on Wed. June 22, 2016 & will continue until completing 8 grabs on Wed. June 22, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to following schedule/QA procedures:

**Wed. June 22:**

**AM Team**

Ensure all test conditions given in Test Day Control Parameters below are set accordingly. **Mention specific existing condition.**

Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

**Starting:**

**After boiler Start-up**

Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

**Ending:**

**After completing 8 grabs:**

Continue 1-minute residue ash grab every hour as per above procedure for 8-hours.

Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (After last sample is collected on 6/21)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>


**Tue. June 28**

07:30–10:00 AM  Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.

Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.

Thank you,

Anil Mebrotra
Plant Engineer
### REPORT OF ANALYSIS

**CLIENT:** Hampton/NASA Steam Plant  
**ATTN:** Anil Mehrotra, Plant Engineer  
**ADDRESS:** 50 Wythe Creek Road  
**CITY:** Hampton, VA 23666  
**PHONE:** (757) 865-1914  
**FAX:** e: amehrotra@hampton.gov  
**SPECIAL NOTES:**

**SAMPLE RECEPT**  
**DATE:** 6/5/2016  
**TIME:** 1055  
**GRAB COLLECTION**  
**DATE:** 6/28/2016  
**TIME:** 0700  
**CLIENT**  
**PICK UP BY:** REED-TL  
**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION ☑️ Good ☐ Other (See C-O-C)**  
**REPORT NO:** 16-11557 11:28

**SAMPLE ID:** HSP-0616-SST12  
**SAMPLE NO:** 16-11557

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1711</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.535</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1711</td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>1.01</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1711</td>
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<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>0.029</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1711</td>
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<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>0.241</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1711</td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt; 0.0002</td>
<td>mg/L</td>
<td>TLG 06/30/16 1648</td>
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<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.050</td>
<td>1</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1711</td>
</tr>
<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1711</td>
</tr>
</tbody>
</table>

---

James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498

VELAP# 460013  
EPA# VA00015
REPORT OF ANALYSIS

NOTES:
JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.
Reproduction of this report is not permitted, except in full, without written approval from James R. Reed & Associates.
The results on this report relate only to the sample(s) provided for analysis.
Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.13
Extraction Fluid used: #2
Final (end point) pH of Extraction Fluid: 5.54

Authorized By: [Signature]
Elaine Claiborne, Laboratory Director
Date: 01-Jul-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax: (757) 873-1498

VELAP# 460013
EPA# VA00015

Page 2 of 2
**ANALYSIS SAMPLE CHAIN OF CUSTODY**

SAMPLE NUMBER: **H5P-0016-SST12**  
MATERIAL: **Residue AS**  
SAMPLE PREPARED BY: **JACK A. DYE**  
METHOD: **H5P-3A**

<table>
<thead>
<tr>
<th>CUSTODIAN</th>
<th>FROM</th>
<th>TO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jack Dye</td>
<td>0700 28 Jun 16</td>
<td>0930 28 Jun 16</td>
</tr>
<tr>
<td>Anna Dye</td>
<td>0930 6/28/16</td>
<td>1030 6/28/16</td>
</tr>
<tr>
<td></td>
<td>1030 6/28/16</td>
<td>1055 6/29/16</td>
</tr>
<tr>
<td></td>
<td></td>
<td>062916</td>
</tr>
</tbody>
</table>

Q. A. Weight: **22.5 lbs**  
Test: **TCLP - all metals**

Reviewed By: [Signature]  
Date: **7/1/16**

Hampton/NASA Steam Plant  
50 Wylde Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
Hampton/NASA Steam Plant
Residue ash Sample Data Records

Sample Certified Valid: 
Sample Date: 6/28/16

<table>
<thead>
<tr>
<th>GRAB</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>1800</td>
<td>1935</td>
<td>2030</td>
<td>2140</td>
<td>2315</td>
<td>2430</td>
<td>0030</td>
</tr>
<tr>
<td>Initials</td>
<td>MB</td>
<td>MB</td>
<td>MB</td>
<td>MB</td>
<td>MB</td>
<td>MB</td>
<td>MB</td>
</tr>
</tbody>
</table>

Date of Collection: 6/22/16

WEIGHT TALLY SHEET (~ or - ½ Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 36.5</td>
<td>15. 3.7</td>
<td>Unburnt Combustibles:</td>
</tr>
<tr>
<td>2. 39.5</td>
<td>16. 4.2</td>
<td>1. 46</td>
</tr>
<tr>
<td>3. 37</td>
<td>17. 46.5</td>
<td>2. 4.55</td>
</tr>
<tr>
<td>4. 42</td>
<td>18. 51</td>
<td>3. 8</td>
</tr>
<tr>
<td>5. 46.5</td>
<td>19. 51.5</td>
<td>4.</td>
</tr>
<tr>
<td>6. 51.5</td>
<td>20. 36.5</td>
<td>5.</td>
</tr>
<tr>
<td>7. 51</td>
<td>21. 15</td>
<td>6.</td>
</tr>
<tr>
<td>8. 36.5</td>
<td>22. 15</td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>9. 15</td>
<td>23. 15</td>
<td>1. 8.5</td>
</tr>
<tr>
<td>10.</td>
<td>24. 1</td>
<td>2.</td>
</tr>
<tr>
<td>11.</td>
<td>25. 1</td>
<td>3.</td>
</tr>
<tr>
<td>13.</td>
<td>27. 1</td>
<td>5.</td>
</tr>
<tr>
<td>14.</td>
<td>28. 1</td>
<td>6.</td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records

(Please Sign and Date below)

Sample # (HSP-3A) HSP-6616-35T12

<table>
<thead>
<tr>
<th>Wet Trench pH:</th>
<th>9.0</th>
<th>Boilers Operating:</th>
<th>1.2</th>
<th>Weather:</th>
<th>Muggy, Rainy</th>
</tr>
</thead>
</table>

Chemical Treatment/ Injection Rate: Sodium Sulphide 12 Gallons/day

Comments: Muggy, Humid, Light Rain at end of test, both boilers were in start-up condition at the time 2 ash samples were collected.

Team(s): TEAM J, ASH TEAM

Quality Leader: Name: JACK DYE  Designation:  

Team Leader: Name:  Designation:  

Mass Proportion Samples

<table>
<thead>
<tr>
<th></th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>855.5</td>
<td>76.8%</td>
<td>15.5 lb</td>
<td>Total: 22.5 lbs. With bucket and lid.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>99.0</td>
<td>21.4%</td>
<td>4.01 lb</td>
<td></td>
</tr>
<tr>
<td>Unburnable</td>
<td>8.5</td>
<td>1.8%</td>
<td>0.5 lb</td>
<td></td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: [Signature]  
Date: 28/5/2016
Hampton/NASA Steam Plant
Residue Ash Sample Treated with \( \text{hp}/\text{gal/day Sod. Sulfide} \)

Method HSP 3A

**Sample # HSP- 0616-SST12** (Both boilers in service)
**Date:** June 28, 2016 (FF inlet Temp. 430 F)

<table>
<thead>
<tr>
<th></th>
<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unburnt</td>
<td>355.5</td>
<td>36.5</td>
<td>46.0</td>
<td>8.5</td>
</tr>
<tr>
<td>Metals</td>
<td>8.5</td>
<td>39.5</td>
<td>45.0</td>
<td>37.0</td>
</tr>
</tbody>
</table>

**Mass Proportion in %**

- Aggregate: 76.8%
- Unburnt: 21.4%
- Metals: 1.8%

<table>
<thead>
<tr>
<th></th>
<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>76.8%</td>
<td>51.5</td>
<td>51.0</td>
<td>100.0%</td>
</tr>
<tr>
<td>Unburnt</td>
<td>21.4%</td>
<td>36.5</td>
<td>15.0</td>
<td></td>
</tr>
<tr>
<td>Metals</td>
<td>1.8%</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Mass Proportion in lbs. for 20 lb. sample**

<table>
<thead>
<tr>
<th></th>
<th>Calculated</th>
<th>Proportion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>15.5</td>
<td>15.36 lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>4.0</td>
<td>4.28 lbs.</td>
</tr>
<tr>
<td>Metals</td>
<td>0.5</td>
<td>0.37 lbs.</td>
</tr>
<tr>
<td>Total</td>
<td>20.0</td>
<td>20.00 lbs.</td>
</tr>
</tbody>
</table>
Hampton/NASA Steam Plant
Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0616–SST13

QA Sample Preparation: Fri. June 24, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to be done during boiler shutdown and start-up on Thu. June 23, 2016 & will continue until completing 8 grabs on Thu. June 23, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to following schedule/QA procedures:

Thu. June 23:

AM Team
Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

During Boiler Shutdown/Start-up Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Ending:
Continue 1-minute residue ash grab every hour as per above procedure for 8-hours.

After completing 8 grabs:
Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (After last sample is collected on 6/22)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>

Remarks: *
1. Mechanical issues
2. Upse. condition: trench cleaning/ wet trash
3. Ash conveyor issues
4. Others

Fri. June 24
07:30–10:00 AM Use Method HSP–3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.
Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.

Thank you,

Anil Mehrotra
Plant Engineer
### Toxic Characteristic Leaching Procedure by SW-846 Method 1311

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Data/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt;0.050</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1706</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.827</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1706</td>
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<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
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<td>mg/L</td>
<td>PEJ 06/30/16 1706</td>
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<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
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<td>5</td>
<td>&lt;0.010</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1706</td>
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</table>
**ANALYSIS SAMPLE CHAIN OF CUSTODY**

**SAMPLE NUMBER:** HSP-0614-55T13  
**MATERIAL:** Residue ASH  
**SAMPLE PREPARED BY:** [Signature]  
**METHOD:** HSP-3A

<table>
<thead>
<tr>
<th>CUSTODIAN</th>
<th>FROM</th>
<th>TO</th>
</tr>
</thead>
<tbody>
<tr>
<td>[Signature]</td>
<td>02/0</td>
<td>24-Jun-16</td>
</tr>
<tr>
<td>[Signature]</td>
<td>1/24</td>
<td>09/5</td>
</tr>
<tr>
<td>[Signature]</td>
<td>5/3</td>
<td>14/03</td>
</tr>
<tr>
<td>[Signature]</td>
<td>6-24</td>
<td>14/00</td>
</tr>
</tbody>
</table>

**Q.A. Weight:** 22.5  
**Test:** TCLP - AN Metals

Reviewed By: [Signature]  
Date: 7/14

Hampton/NASA Steam Plant  
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

<table>
<thead>
<tr>
<th>SAMPLE #</th>
<th>Sample Date:</th>
<th>Method Used:</th>
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<tbody>
<tr>
<td>HS0-0616-SS13</td>
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<td>HS 3A</td>
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Sample Certified Valid:

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<tr>
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<td>1031</td>
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<table>
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<tr>
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<th>11</th>
<th>12</th>
<th>13</th>
<th>14</th>
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<th>16</th>
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</thead>
<tbody>
<tr>
<td>Time</td>
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</tr>
<tr>
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<td></td>
</tr>
</tbody>
</table>

---

### WEIGHT TALLY SHEET (+ or - ½ Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 40.5</td>
<td>15.</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 41.5</td>
<td>16.</td>
<td>1. 13.5</td>
</tr>
<tr>
<td>3. 44</td>
<td>17.</td>
<td>2. 35</td>
</tr>
<tr>
<td>4. 53</td>
<td>18.</td>
<td>3.</td>
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<td>5. 46.5</td>
<td>19.</td>
<td>4.</td>
</tr>
<tr>
<td>6. 47</td>
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<td>5.</td>
</tr>
<tr>
<td>7. 48.5</td>
<td>21.</td>
<td>6.</td>
</tr>
<tr>
<td>8. 37.5</td>
<td>22.</td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>9.</td>
<td>23.</td>
<td>1. 10</td>
</tr>
<tr>
<td>10.</td>
<td>24.</td>
<td>2.</td>
</tr>
<tr>
<td>11.</td>
<td>25.</td>
<td>3.</td>
</tr>
<tr>
<td>12.</td>
<td>26.</td>
<td>4.</td>
</tr>
<tr>
<td>13.</td>
<td>27.</td>
<td>5.</td>
</tr>
<tr>
<td>14.</td>
<td>28.</td>
<td>6.</td>
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</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample #: HSP-0616-SST13

<table>
<thead>
<tr>
<th>Wet Trench pH:</th>
<th>8.3</th>
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</thead>
<tbody>
<tr>
<td>Boilers Operating:</td>
<td>1/2</td>
</tr>
<tr>
<td>Weather:</td>
<td>Rainy/Thunder</td>
</tr>
<tr>
<td>Chemical Treatment/ Injection Rate:</td>
<td></td>
</tr>
</tbody>
</table>

Comments: Rainy, Thunder, Power Failure
- First Grab: Very little residue came out
- 1/4 Grab for burn down
- 1000 Grab: Very/Very little Water also for 1031 Grab
- Switched from Trench 2 to Trench 1. Dugged Trench 2
- Sodium Sulfide Rate: 10 g/day

Team (s):

Quality Leader: Name Jack Dye  Designation: AP

Team Leader: Name  Designation: 

<table>
<thead>
<tr>
<th>Mass Proportion Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lbs.</td>
</tr>
<tr>
<td>------</td>
</tr>
<tr>
<td>Aggregate</td>
</tr>
<tr>
<td>Unburnt</td>
</tr>
<tr>
<td>Unburnable</td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: Jack Dye

Date: 24-WN16
Calculation Sheet

20 lb. Ash Sample

Sample #: HSP-0016-SJT13

Date: 6/24/2016

1. Sub Total (lbs)
   a. Aggregate 348.5
   b. Unburnt 48.5
   c. Metal 10.0
   d. Total 407.0

2. Mass Proportion (%)
   a. Aggregate \( \frac{a}{d} \times 100\% \) 85.62
   b. Unburnt \( \frac{b}{d} \times 100\% \) 11.91
   c. Metal \( \frac{c}{d} \times 100\% \) 2.45
   d. Total 100%

3. Calculate 20 lb Proportion (lb)
   (upto 2 decimal places)
   Aggregate \( (A \times 20 lb) \) 17.12
   Unburnt \( (B \times 20 lb) \) 2.38
   Metal \( (C \times 20 lb) \) 4.9
   Total 20 lb

4. Rounded Up/Down (lb)
   (upto 1/2 lb)
   Aggregate 17.0 lb.
   Unburnt 2.5 lb.
   Metal 0.5 lb.
   Total 20.0 lb.
Hampton/NASA Steam Plant
Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0616–SST14

Sample Collection: Mon. June 27, 2016
QA Sample Preparation: Tue. June 28, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon on Mon. June 27, 2016 & will continue until 7:00 pm on Mon. June 27, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to following schedule/QA procedures:

Mon. June 27:
AM Team
Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Starting:
12:00 noon
Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Ending:
19:00 hrs
Continue 1-minute residue ash grab every hour as per above
Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (After last sample is collected on 6/23)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Reagent Specific Gravity</strong></td>
</tr>
<tr>
<td><strong>Fabric Filter Inlet Temp.</strong></td>
</tr>
<tr>
<td><strong>Boilers steam rate</strong></td>
</tr>
<tr>
<td><strong>Boilers running condition</strong></td>
</tr>
</tbody>
</table>

Tue. June 28
07:30–10:00 AM  Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.
Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.

Thank you,

Anil Mehrotra
Plant Engineer
## REPORT OF ANALYSIS

**CLIENT:** Hampton/NASA Steam Plant  
**SAMPLE RECEIPT**  
**ATTN:** Anil Mehrotra, Plant Engineer  
**DATE:** 6/29/2016  
**ADDRESS:** 50 Wythe Creek Road  
**TIME:** 1055  
**CITY:** Hampton, VA 23666  
**PHONE:** (757) 865-1914  
**FAX:** c: amehrotra@hampton.gov  
**COLLECTED BY:** CLIENT  
**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION ☑ Good ☐ Other (See C-O-C)**  
**PICK UP BY:** REED-TL  
**REPORT NO:** 16-11558 11:28

**SAMPLE ID:** HSP-0616-SST/14  
**SAMPLE NO:** 16-11558

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt;0.050</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1515</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.845</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1515</td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.253</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1515</td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt;0.010</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1515</td>
</tr>
<tr>
<td>Lead</td>
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<td>6010C</td>
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<td>0.112</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1515</td>
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<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt;0.0002</td>
<td>mg/L</td>
<td>TLG 06/30/16 1724</td>
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<td>Selenium</td>
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<td>6010C</td>
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<td>1</td>
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<tr>
<td>Silver</td>
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<td>6010C</td>
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<td>5</td>
<td>&lt;0.010</td>
<td>mg/L</td>
<td>PEJ 06/30/16 1515</td>
</tr>
</tbody>
</table>

---

James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23605  
(757) 873-4703  Fax: (757) 873-1498  
VELAP# 460013  
EPA# VA00015

Page 1 of 2
REPORT OF ANALYSIS

NOTES:

JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.

Reproduction of this report is not permitted, except in full, without written approval from James R Reed & Associates.

The results on this report relate only to the sample(s) provided for analysis.

Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.75
Extraction Fluid used: #1
Final (end point) pH of Extraction Fluid: 7.39

Authorized By: [Signature]
Elaine Claiborne, Laboratory Director
Date: 01-Jul-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4701 • Fax: (757) 873-1498

VELAP# 460013
EPA# VA00015
## ANALYSIS SAMPLE CHAIN OF CUSTODY

**SAMPLE NUMBER:** HSP-0616-5ST14  
**MATERIAL:** Resin 1541  
**SAMPLE PREPARED BY:** Jack P. Ray  
**METHOD:** HSP-3A

### Custodian Log

<table>
<thead>
<tr>
<th>Custodian</th>
<th>From</th>
<th>To</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jack P. Ray</td>
<td>0000</td>
<td>1030</td>
<td>28 June 16</td>
</tr>
<tr>
<td>Jack P. Ray</td>
<td>1030</td>
<td>17816</td>
<td>28 June 16</td>
</tr>
<tr>
<td>John Green</td>
<td>1030</td>
<td>062916</td>
<td>01/29/16</td>
</tr>
<tr>
<td>John Green</td>
<td>1055</td>
<td>062916</td>
<td></td>
</tr>
</tbody>
</table>

Q. A. Weight: 225.66  
Test: TELP: H2 Me Metals

Reviewed By: [Signature]  
Date: 7/1/16

Hampton/NASA Steam Plant  
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

<table>
<thead>
<tr>
<th>SAMPLE #</th>
<th>150 06/16-55714</th>
<th>Sample Date: 6/128/76</th>
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Sample Certified Valid:

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<td>Initials</td>
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<td></td>
</tr>
</tbody>
</table>

### WEIGHT TALLY SHEET (+ or - 1/2 Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 41</td>
<td>15. 43</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 43</td>
<td>16.</td>
<td>1. 56.5</td>
</tr>
<tr>
<td>3. 41.5</td>
<td>17.</td>
<td>2. 52.5</td>
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<tr>
<td>4. 39.5</td>
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<td>3. 42.5</td>
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<tr>
<td>5. 40.5</td>
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<td>5.</td>
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<tr>
<td>7. 44.5</td>
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<td>6.</td>
</tr>
<tr>
<td>8. 47</td>
<td>22.</td>
<td></td>
</tr>
<tr>
<td>9. 44</td>
<td>23.</td>
<td></td>
</tr>
<tr>
<td>10. 43</td>
<td>24.</td>
<td></td>
</tr>
<tr>
<td>11. 40.5</td>
<td>25.</td>
<td></td>
</tr>
<tr>
<td>13. 44</td>
<td>27.</td>
<td></td>
</tr>
<tr>
<td>14. 44</td>
<td>28.</td>
<td></td>
</tr>
<tr>
<td>15. 43</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Unburnable (Metal)

| 1. 13.5 |
| 2.      |
| 3.      |
| 4.      |
| 5.      |
| 6.      |
Residue ash Sample Data Records  
(Please Sign and Date below)

Sample # 1602016-55714

<table>
<thead>
<tr>
<th>Wet Trench pH: 8.7</th>
<th>Boilers Operating: 18.2</th>
<th>Weather: Cloudy, Light Rain</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Treatment/Injection Rate: Sodium Hydroxide injection. 10/11 Day</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Team(s):

Quality Leader Name: Jack A. Age
Designation:______

Team Leader Name:______
Designation:______

<table>
<thead>
<tr>
<th>Mass Proportion Samples</th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>647</td>
<td>76.7</td>
<td>15.5</td>
<td>Total: 22.5 Lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>182.5</td>
<td>21.6</td>
<td>4</td>
<td>With bucket and lid.</td>
</tr>
<tr>
<td>Unburnable</td>
<td>13.5</td>
<td>1.6</td>
<td>0.5</td>
<td></td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: [Signature]
Date: 28 Jun 16
**Hampton/NASA Steam Plant**

Residue Ash Sample Treated with Hydroxylamine Sulfide

Sample collected on June 27, 2016

**Method HSP 3A**

**Sample # HSP-0616-SST14** (Both boilers in service)

**Date: June 28, 2016** (FF inlet Temp. 430 F)

<table>
<thead>
<tr>
<th></th>
<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Aggregate</strong></td>
<td>647.0</td>
<td>41.0</td>
<td>56.5</td>
</tr>
<tr>
<td><strong>Unburnt</strong></td>
<td>182.5</td>
<td>43.0</td>
<td>52.5</td>
</tr>
<tr>
<td><strong>Metals</strong></td>
<td>13.5</td>
<td>41.5</td>
<td>42.5</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>843.0</td>
<td>39.5</td>
<td>31.0</td>
</tr>
</tbody>
</table>

**Mass Proportion in %**

<table>
<thead>
<tr>
<th></th>
<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Aggregate</strong></td>
<td>76.7%</td>
<td>39.5</td>
<td></td>
</tr>
<tr>
<td><strong>Unburnt</strong></td>
<td>21.6%</td>
<td>44.5</td>
<td></td>
</tr>
<tr>
<td><strong>Metals</strong></td>
<td>1.6%</td>
<td>47.0</td>
<td></td>
</tr>
<tr>
<td><strong>100.0%</strong></td>
<td></td>
<td>44.0</td>
<td>43.0</td>
</tr>
<tr>
<td><strong>40.5</strong></td>
<td></td>
<td>40.5</td>
<td>47.0</td>
</tr>
</tbody>
</table>

**Mass Proportion in lbs. for 20 lb. sample**

<table>
<thead>
<tr>
<th></th>
<th>Calculated</th>
<th>Proportion</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Aggregate</strong></td>
<td>15.5</td>
<td>15.35 lbs.</td>
</tr>
<tr>
<td><strong>Unburnt</strong></td>
<td>4.0</td>
<td>4.33 lbs.</td>
</tr>
<tr>
<td><strong>Metals</strong></td>
<td>0.5</td>
<td>0.32 lbs.</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>20.0</td>
<td>20.00 lbs.</td>
</tr>
</tbody>
</table>
Hampton/NASA Steam Plant
Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0616–SST15

Sample Collection: Tue. June 28, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon on Tue. June 28, 2016 & will continue until 7:00 pm on Tue. June 28, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to following schedule/QA procedures:

Tue. June 28:
AM Team
Starting:
12:00 noon
Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Ending:
19:00 hrs:
Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Continue 1-minute residue ash grab every hour as per above
Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

| Test Day Control Parameters (After last sample is collected on 6/27) |
|--------------------------|----------------|-----------|
| Reagent Specific Gravity | 1.01           | SO₂ Control | 60 ppm  |
| Fabric Filter Inlet Temp. | 430°F         | FF Modules on | All 3   |
| Boilers steam rate       | 33 kpph       | Sodium Sulfide rate | 300 g/day |
| Boilers running condition | Normal: Y/N * | Any important change |


Wed. June 29
07:30–10:00 AM Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample. Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297. Thank you.

Anil Mehrotra
Plant Engineer
### REPORT OF ANALYSIS

**CLIENT:** Hampton/NASA Steam Plant  
**SAMPLE RECEIPT**

**ATTN:** Anil Mehrotra, Plant Engineer  
**DATE:** 7/5/2016  
**TIME:** 1230

**ADDRESS:** 50 Wythe Creek Road  
**GRAB COLLECTION**

**CITY:** Hampton, VA 23666  
**DATE:** 6/29/2016  
**TIME:** 0000

**PHONE:** (757) 865-1914  
**COLLECTED BY:** CLIENT

**FAX:** e: amehrotra@hampton.gov  
**PICK UP BY:** REED - AC

**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION:** ✔️ Good

**SPECIAL NOTES:**  
**REPORT NO:** 16-11732 14:42

---

**SAMPLE ID:** HSP-0616-SST15-A  
**SAMPLE NO:** 16-11732

---

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA Level</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ</td>
<td>07/08/16 1517</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.641</td>
<td>mg/L</td>
<td>PEJ</td>
<td>07/08/16 1517</td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.167</td>
<td>mg/L</td>
<td>PEJ</td>
<td>07/08/16 1517</td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ</td>
<td>07/08/16 1517</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ</td>
<td>07/08/16 1517</td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>2</td>
<td>&lt; 0.0002</td>
<td>mg/L</td>
<td>TLG</td>
<td>07/07/16 1025</td>
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<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.050</td>
<td>1</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ</td>
<td>07/08/16 1517</td>
</tr>
<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ</td>
<td>07/08/16 1517</td>
</tr>
</tbody>
</table>

---

James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703  ● Fax: (757) 873-1498

VELAP#: 460013  
EPA#: VA000015

Page 1 of 2
NOTES:
JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.

Reproduction of this report is not permitted, except in full, without written approval from James R. Reed & Associates.

The results on this report relate only to the sample(s) provided for analysis.

Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.66
Extraction Fluid used: 1
Final (end point) pH of Extraction Fluid: 8.52

Authorized By: [Signature]
Elaine Claiborne, Laboratory Director
Date: 11-Jul-16
**ANALYSIS SAMPLE**  
**CHAIN OF CUSTODY**

**SAMPLE NUMBER:** HSP-01655-T15-A  
**MATERIAL:** Residue Am

**SAMPLE PREPARED BY:** Donald N.  
**METHOD:** HSP-2A

<table>
<thead>
<tr>
<th>CUSTODIAN</th>
<th>FROM</th>
<th>TO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Donald N.</td>
<td>6/29/16</td>
<td>0700</td>
</tr>
<tr>
<td></td>
<td>6/29/16</td>
<td>0855</td>
</tr>
<tr>
<td></td>
<td>7/5/16</td>
<td>12:15</td>
</tr>
<tr>
<td></td>
<td>7-5-16</td>
<td>1015</td>
</tr>
<tr>
<td></td>
<td>7-5-16</td>
<td>1230</td>
</tr>
<tr>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Q. A. Weight:** 22.5 lb  
**Test:** TCLP All Metals

Reviewed By: [Signature]

Date: 7/15/16

Hampton/NASA Steam Plant
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

<table>
<thead>
<tr>
<th>SAMPLE #</th>
<th>Sample Date</th>
<th>Method Used</th>
</tr>
</thead>
<tbody>
<tr>
<td>HSP-0610</td>
<td>7/15</td>
<td>HSP-3A</td>
</tr>
</tbody>
</table>

Sample Certified Valid: [Signature]

<table>
<thead>
<tr>
<th>GRAB</th>
<th>28/June</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>12:03</td>
<td>13:07</td>
<td>14:19</td>
<td>15:02</td>
<td>16:15</td>
<td>17:15</td>
<td>18:05</td>
<td>19:15</td>
</tr>
<tr>
<td>Initials</td>
<td>DAD</td>
<td>MPP</td>
<td>MPP</td>
<td>MPP</td>
<td>MPP</td>
<td>MPP</td>
<td>MPP</td>
<td>MPP</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>GRAB</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
<th>13</th>
<th>14</th>
<th>15</th>
<th>16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initials</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>GRAB</th>
<th>17</th>
<th>18</th>
<th>19</th>
<th>20</th>
<th>21</th>
<th>22</th>
<th>23</th>
<th>24</th>
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</thead>
<tbody>
<tr>
<td>Time</td>
<td></td>
<td></td>
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<td>Initials</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### WEIGHT TALLY SHEET (+ or - ½ Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 47.5</td>
<td>15.</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 44.5</td>
<td>16.</td>
<td>1. 32</td>
</tr>
<tr>
<td>3. 36.5</td>
<td>17.</td>
<td>2. 35</td>
</tr>
<tr>
<td>4. 22</td>
<td>18.</td>
<td>3. 30.5</td>
</tr>
<tr>
<td>5. 42.5</td>
<td>19.</td>
<td>4. 35</td>
</tr>
<tr>
<td>6. 40</td>
<td>20.</td>
<td>5. Unburnable (Metal)</td>
</tr>
<tr>
<td>7. 38</td>
<td>21.</td>
<td>6.</td>
</tr>
<tr>
<td>8. 31.5</td>
<td>22.</td>
<td></td>
</tr>
<tr>
<td>9. 41</td>
<td>23.</td>
<td></td>
</tr>
<tr>
<td>10. 45.5</td>
<td>24.</td>
<td></td>
</tr>
<tr>
<td>11. 30.5</td>
<td>25.</td>
<td></td>
</tr>
<tr>
<td>13.</td>
<td>27.</td>
<td></td>
</tr>
<tr>
<td>14.</td>
<td>28.</td>
<td></td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample #: HSP-1110-3ST 15

<table>
<thead>
<tr>
<th>Wet Trench pH: 9.1</th>
<th>Boilers Operating: 142</th>
<th>Weather: clear</th>
</tr>
</thead>
</table>

Chemical Treatment/ Injection Rate: Sodium Sulphate, Iron Oxide

Comments:

Team (s): A. Smith, R. Davidson

Quality Leader: Name: Jack Dye  Designation: Ash Handler

Team Leader: Name: David Wilson  Designation: OP ENL

<table>
<thead>
<tr>
<th>Mass Proportion Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lbs.</td>
</tr>
<tr>
<td>------</td>
</tr>
<tr>
<td>Aggregate</td>
</tr>
<tr>
<td>Unburnt</td>
</tr>
<tr>
<td>Unburnable</td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: [Signature]

Date: 6/29/16
Hampton/NASA Steam Plant
Residue Ash Sample Treated with 16 gal/day Sod. Sulfide
Sample collected on June 28, 2016
Method HSP 3A

Sample # HSP- 0616-SST15 (Both boilers in service)
Date: June 29, 2016 (FF inlet Temp. 430 F)

<table>
<thead>
<tr>
<th></th>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Unburnt</td>
<td>132.5</td>
<td>47.5</td>
<td>32.0</td>
<td>7.5</td>
<td></td>
</tr>
<tr>
<td>Metals</td>
<td>7.5</td>
<td>44.5</td>
<td>35.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>559.5</td>
<td>36.5</td>
<td>30.5</td>
<td>22.0</td>
<td>35.0</td>
</tr>
</tbody>
</table>

**Mass Proportion in %**

<table>
<thead>
<tr>
<th></th>
<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>75.0%</td>
<td>40.0</td>
<td>38.0</td>
<td>42.5</td>
</tr>
<tr>
<td></td>
<td>23.7%</td>
<td>31.5</td>
<td>41.0</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.3%</td>
<td>100.0%</td>
<td>45.5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>100.0%</td>
<td></td>
<td>30.5</td>
<td></td>
</tr>
</tbody>
</table>

**Mass Proportion in lbs. for 20 lb. sample**

<table>
<thead>
<tr>
<th></th>
<th>Calculated in lbs.</th>
<th>Proportion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>15.0</td>
<td>15.00 lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>4.5</td>
<td>4.74 lbs.</td>
</tr>
<tr>
<td>Metals</td>
<td>0.5</td>
<td>0.27 lbs.</td>
</tr>
<tr>
<td>Total</td>
<td>20.0</td>
<td>20.00 lbs.</td>
</tr>
</tbody>
</table>
Hampton/NASA Steam Plant
Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0716–SST16


An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon on Wed. July 27, 2016 & will continue until 7:00 pm on Wed. July 27, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to following schedule/QA procedures:

Wed. July 27:
AM Team
Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Starting:
12:00 noon
Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Ending:
19:00 hrs
Continue 1-minute residue ash grab every hour as per above
Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (Set these conditions before collecting samples)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>


Thu. July 28
07:30–10:00 AM Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.
Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.
Thank you,
Anil Mehrotra
Plant Engineer

[Chemical discharge rate calculations and measurements]
# REPORT OF ANALYSIS

**CLIENT:** Hampton NASA Steam Plant  
**SAMPLE RECEIVED**

**ATTN:** Anil Mehertra, Plant Engineer  
**DATE:** 7/29/2016  
**TIME:** 13:15

**ADDRESS:** 50 Wythe Creek Road  
**GRAB COLLECTION**

**CITY:** Hampton, VA 23666  
**DATE:** 7/28/2016  
**TIME:** 00:00

**PHONE:** (757) 865-1914  
**COLLECTED BY:** CLIENT

**FAX:** cmehertra@hampton.gov  
**PICK UP BY:** REED - ML

**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION** Good  
**Other (See C-O-C)**

**REPORT NO:** 16-13465 15:29

---

**SAMPLE ID:** HSP-0716-SST16  
**SAMPLE NO:** 16-13465

---

**Parameter**  
**Toxic Characteristic Leaching Procedure by SW-846 Method 1311**  

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA Number</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 08/05/16 1224</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.350</td>
<td>mg/L</td>
<td>PEJ 08/05/16 1224</td>
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<tr>
<td>Cadmium</td>
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<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.420</td>
<td>mg/L</td>
<td>PEJ 08/05/16 1224</td>
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<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 08/05/16 1224</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>0.095</td>
<td>mg/L</td>
<td>PEJ 08/05/16 1224</td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt; 0.0002</td>
<td>mg/L</td>
<td>TLG 08/05/16 1159</td>
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<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
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<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 08/05/16 1224</td>
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<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 08/05/16 1224</td>
</tr>
</tbody>
</table>

---

James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498  
VELAP# 460013  
EPA# VA00015  
Page 1 of 2
REPORT OF ANALYSIS

NOTES:

NRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.
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The results on this report relate only to the sample(s) provided for analysis.
Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.25
Extraction Fluid used: 2
Final (end point) pH of Extraction Fluid: 5.95

Authorized By: [Signature]
Elaine Claiborne, Laboratory Director
Date: 05-Aug-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax: (757) 873-1498
# Analysis Sample Chain of Custody

**Sample Number:** HSP-0716-SS716  
**Material:** Residue Ash  
**Sample Prepared By:** [Signature]  
**Method:** HSP-3A

<table>
<thead>
<tr>
<th>Custodian</th>
<th>From</th>
<th>To</th>
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</thead>
<tbody>
<tr>
<td>[Signature]</td>
<td>07/04</td>
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<tr>
<td>[Signature]</td>
<td>9.41</td>
<td>29/July</td>
</tr>
<tr>
<td>[Signature]</td>
<td>07/03</td>
<td>7/29/16</td>
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</tbody>
</table>

Q.A. Weight: 22.5 lbs  
Test: TCLP  
All Metals  
Rush Results

Reviewed By: [Signature]  
Date: 8/5/16

Hampton/NASA Steam Plant  
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

<table>
<thead>
<tr>
<th>SAMPLE #</th>
<th>HSP-0716-55714</th>
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</thead>
<tbody>
<tr>
<td>Method Used</td>
<td>HSP-3A</td>
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<tr>
<td>Sample Certified Valid:</td>
<td>And Melvyn, 8/6/16</td>
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<table>
<thead>
<tr>
<th>GRAB</th>
<th>2</th>
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<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
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<tbody>
<tr>
<td>Time</td>
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<td>130</td>
<td>140</td>
<td>150</td>
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<td>JDA</td>
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<td>JDA</td>
<td>JDA</td>
<td>JDA</td>
<td>JDA</td>
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<thead>
<tr>
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<th>11</th>
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<th>13</th>
<th>14</th>
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<tbody>
<tr>
<td>Time</td>
<td>JDA</td>
<td>JDA</td>
<td>JDA</td>
<td>JDA</td>
<td>JDA</td>
<td>JDA</td>
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<tr>
<td>Initials</td>
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<td>JDA</td>
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<td>JDA</td>
<td>JDA</td>
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</table>

### WEIGHT TALLY SHEET (+ or - ¼ Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.  31</td>
<td>15. 47</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2.  38½</td>
<td>16. 44½</td>
<td>1. 22½</td>
</tr>
<tr>
<td>3.  43</td>
<td>17. 33</td>
<td>2.</td>
</tr>
<tr>
<td>4.  46</td>
<td>18. 20½</td>
<td>3.</td>
</tr>
<tr>
<td>5.  37½</td>
<td>19.</td>
<td></td>
</tr>
<tr>
<td>6.  37</td>
<td>20.</td>
<td></td>
</tr>
<tr>
<td>7.  36½</td>
<td>21.</td>
<td></td>
</tr>
<tr>
<td>8.  37½</td>
<td>22.</td>
<td></td>
</tr>
<tr>
<td>9.  46</td>
<td>23.</td>
<td></td>
</tr>
<tr>
<td>10. 44</td>
<td>24.</td>
<td></td>
</tr>
<tr>
<td>11. 43</td>
<td>25.</td>
<td></td>
</tr>
<tr>
<td>12. 45</td>
<td>26.</td>
<td></td>
</tr>
<tr>
<td>13. 49½</td>
<td>27.</td>
<td></td>
</tr>
<tr>
<td>14. 44</td>
<td>28.</td>
<td></td>
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</tbody>
</table>

Unburnable (Metal)
Residue ash Sample Data Records
(Please Sign and Date below)

Sample # HSP-0716-55176

<table>
<thead>
<tr>
<th>Wet Trench pH: 10.1</th>
<th>Boilers Operating: 142</th>
<th>Weather: Hot, Muggy</th>
</tr>
</thead>
</table>

Chemical Treatment/Injection Rate: Sodium Sulfide

Comments: 1.8 3, 4 gallons/day

Pump #1 set at 17 strokes/min.

Chemical discharge measured

at Fly Ash Decker

= 30 gal/min

= 4.4 gal/day

13.3

Team(s): B

Quality Leader: Name: Jack D. Dye  Designation: AB

Team Leader: Name: Phil Gamble  Designation: CE

Mass Proportion Samples

<table>
<thead>
<tr>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>78.5</td>
<td>18.5</td>
<td>9.22</td>
</tr>
<tr>
<td>Unburnt</td>
<td>22.6</td>
<td>0.5</td>
<td>0.07</td>
</tr>
<tr>
<td>Unburnable</td>
<td>22</td>
<td>0.5</td>
<td>0.13</td>
</tr>
</tbody>
</table>

Total: 82.5 Lbs. With bucket and lid.

Verification by Quality Leader or Team Leader

Sign: [Signature]  Date: 23 July 16
Hampton/NASA Steam Plant
Residue Ash Sample Treated with 11,4 gal/day Sod. Sulfide
Sample collected on July 27, 2016
Method HSP 3A

Sample # HSP- 0716-SST16 (Both boilers in service)
Date: July 28, 2016 (FF inlet Temp. 430 F)

<table>
<thead>
<tr>
<th></th>
<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unburnt</td>
<td>22.5</td>
<td>31.0</td>
<td>22.5</td>
</tr>
<tr>
<td>Metals</td>
<td>22.0</td>
<td>38.5</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>768.0</td>
<td>43.0</td>
<td>46.0</td>
</tr>
</tbody>
</table>

**Mass Proportion in %**
<table>
<thead>
<tr>
<th></th>
<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>94.2%</td>
<td>37.0</td>
<td></td>
</tr>
<tr>
<td>Unburnt</td>
<td>2.9%</td>
<td>36.5</td>
<td></td>
</tr>
<tr>
<td>Metals</td>
<td>2.9%</td>
<td>37.5</td>
<td></td>
</tr>
<tr>
<td>100.0%</td>
<td>46.0</td>
<td>44.0</td>
<td>43.0</td>
</tr>
<tr>
<td></td>
<td>45.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Mass Proportion in lbs. for 20 lb. sample**
<table>
<thead>
<tr>
<th></th>
<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>49.5</td>
<td>44.0</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Rounded Up Calculated in lbs. Proportion**
<table>
<thead>
<tr>
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<th>Aggregate</th>
<th>Unburnt</th>
<th>Metals</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>19.0</td>
<td>18.84 lbs.</td>
<td>33.0</td>
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<tr>
<td></td>
<td>0.5</td>
<td>0.59 lbs.</td>
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<tr>
<td></td>
<td>0.5</td>
<td>0.57 lbs.</td>
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</tr>
<tr>
<td>Total</td>
<td>20.0</td>
<td>20.00 lbs.</td>
<td></td>
</tr>
</tbody>
</table>
Hampton/NASA Steam Plant
Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0316–SST17


An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon on Thu. Aug 4, 2016 & will continue until 7:00 pm on Thu. Aug 4, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to following schedule/QA procedures:

Thu. Aug 4th
AM Team
Starting: 12:00 noon
Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Ending: 19:00 hrs
Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (Set these conditions before collecting samples)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>


Fri. Aug 5
07:30–10:00 AM Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.
Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.

Thank you,

Anil Mehrotra
Plant Engineer
# REPORT OF ANALYSIS

**CLIENT:** Hampton/NASA Steam Plant  
**ATTN:** Anil Mehrotra, Plant Engineer  
**ADDRESS:** 50 Wythe Creek Road  
**CITY:** Hampton, VA 23666  
**PHONE:** (757) 865-1914  
**FAX:** c: amehrotra@hampton.gov  

**SAMPLE RECEIPT**  
**DATE:** 8/2/2016  
**TIME:** 13:15  
**GRAB COLLECTION**  
**DATE:** 8/5/2016  
**TIME:** 00:00  
**COLLECTED BY:** CLIENT  
**PICK UP BY:** REED - BS  
**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION** ✔️ Good  

**REPORT NO:** 16-13911  
**15 sukes/min**  
**12.2 µg/d**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>TLG</th>
<th>08/12/16</th>
<th>1445</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1445</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.429</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1445</td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>&lt; 0.906</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1445</td>
</tr>
<tr>
<td>Chromium</td>
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<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1445</td>
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<tr>
<td>Lead</td>
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<td>6010C</td>
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<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1445</td>
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<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>0.2</td>
<td>&lt; 0.0002</td>
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<td>&lt; 0.050</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1445</td>
</tr>
<tr>
<td>Silver</td>
<td>D011</td>
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<td>&lt; 0.010</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1445</td>
</tr>
</tbody>
</table>

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(757) 873-4703  •  Fax: (757) 873-1498  
VELAP# 460013  
EPAA VA00015  
Page 1 of 2
REPORT OF ANALYSIS

NOTES:

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Results conform to HELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.62
Extraction Fluid used: 2
Final (and point) pH of Extraction Fluid: 5.77

M. Matrix spike % recovery outside acceptance range (71%)
   possibility due to high background.

Authorized By: Elaine Claiborne, Laboratory Director
Date: 12-Aug-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax: (757) 873-1498

VELAP# 460013
EPA# VA00015
### REPORT OF ANALYSIS

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
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<td>D006</td>
<td>6010C</td>
<td>0.003</td>
<td>1</td>
<td>0.906 M</td>
<td>mg/L</td>
<td>08/12/16 1445</td>
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<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
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<td>mg/L</td>
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</tr>
<tr>
<td>Mercury</td>
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<td>7470A</td>
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<td>mg/L</td>
<td>08/11/16 1208</td>
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<td>mg/L</td>
<td>08/12/16 1445</td>
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<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>08/12/16 1445</td>
</tr>
</tbody>
</table>

**Date:** Aug 5, 16  
**Pump Setting:** 15 gpm  
**SSI Rate:** 12 gpm/day  
**Results:** Sat
# Analysis Sample Chain of Custody

**Sample Number:** HSP-0816-55717  
**Material:** Residue Ash  
**Sample Prepared By:** [Signature]  
**Method:** HSP-3A  

<table>
<thead>
<tr>
<th>Custodian</th>
<th>From</th>
<th>To</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>John</td>
<td>0701</td>
<td>0755</td>
<td>5th Aug 16</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1250</td>
</tr>
<tr>
<td>ES</td>
<td>8/116</td>
<td>1315</td>
<td>8/5/16</td>
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<tr>
<td>CBAR</td>
<td></td>
<td>1315</td>
<td></td>
</tr>
</tbody>
</table>

**Q. A. Weight:** 22.5  
**Test:** TCLP  
All Metals  
Rush Results

---

Hampton/NASA Steam Plant  
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
<table>
<thead>
<tr>
<th>SAMPLE #</th>
<th>HSP-0816-5ST7</th>
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<tr>
<td>Method Used</td>
<td></td>
<td>8/12/16 Plant Engineer</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>GRAB</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
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<tr>
<td>Time</td>
<td>1205</td>
<td>1302</td>
<td>1404</td>
<td>1505</td>
<td>1602</td>
<td>1700</td>
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<td>1909</td>
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<td>12</td>
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<td>14</td>
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<td>Time</td>
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</tr>
<tr>
<td>GRAB</td>
<td>17</td>
<td>18</td>
<td>19</td>
<td>20</td>
<td>21</td>
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<td>Time</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Initials</td>
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<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>

### WEIGHT TALLY SHEET (+ or - ¼ Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2″</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 36½</td>
<td>15. 28</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 49</td>
<td>16. 28½</td>
<td></td>
</tr>
<tr>
<td>3. 45½</td>
<td>17. 26</td>
<td></td>
</tr>
<tr>
<td>4. 48½</td>
<td>18. 22</td>
<td></td>
</tr>
<tr>
<td>5. 45</td>
<td>19. 24</td>
<td></td>
</tr>
<tr>
<td>6. 51½</td>
<td>20.</td>
<td></td>
</tr>
<tr>
<td>7. 28½</td>
<td>21. Total 705.5</td>
<td></td>
</tr>
<tr>
<td>8. 42</td>
<td>22.</td>
<td></td>
</tr>
<tr>
<td>9. 37</td>
<td>23.</td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>10. 33½</td>
<td>24.</td>
<td></td>
</tr>
<tr>
<td>11. 31½</td>
<td>25.</td>
<td></td>
</tr>
<tr>
<td>13. 28½</td>
<td>27.</td>
<td></td>
</tr>
<tr>
<td>15. 28</td>
<td></td>
<td>1. 14</td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample #: HNSP-0816 - SST17

<table>
<thead>
<tr>
<th>Wet Trench pH</th>
<th>Boilers Operating</th>
<th>Weather</th>
<th>Chemical Treatment/Injection Rate</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.1</td>
<td>#1, #2</td>
<td>Part Cloudy</td>
<td>15 strokes/min 12gal/day</td>
<td>1500 g/hour Super Bin Plugged up. Top Rate Valve Stopped for short time, main wheel both open</td>
</tr>
</tbody>
</table>

Team(s):

Quality Leader: Name Jack Dye  Designation: AO

Team Leader: Name Martin Benfield  Designation: OF

<table>
<thead>
<tr>
<th>Mass Proportion Samples</th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>670.5</td>
<td>95.03</td>
<td><strong>519.0 lbs</strong></td>
<td>Total: <strong>22.5</strong> Lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>21.0</td>
<td>2.97</td>
<td>.5 lbs</td>
<td>With bucket and lid.</td>
</tr>
<tr>
<td>Unburnable</td>
<td>14.0</td>
<td>1.98</td>
<td>.5 lbs</td>
<td></td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: [Signature]
Date: 8/5/16
## Calculation Sheet

**Sample: 20 lb. Ash Sample**

**Sample #:** HSP-0816-5ST 17

**Date:** Aug. 5, 2016

<table>
<thead>
<tr>
<th>1. Sub Total (Lbs)</th>
<th>2. Mass Proportion (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Aggregate 670.5</td>
<td>a. Aggregate ( \frac{A}{D} \times 100% ) - 95.03</td>
</tr>
<tr>
<td>b. Unburnt 21.0</td>
<td>b. Unburnt ( \frac{B}{D} \times 100% ) - 2.97</td>
</tr>
<tr>
<td>c. Metal 14.0</td>
<td>c. Metal ( \frac{C}{D} \times 100% ) - 1.99</td>
</tr>
<tr>
<td>d. <strong>TOTAL</strong> 705.5</td>
<td><strong>D. Total</strong> 100%</td>
</tr>
</tbody>
</table>

### Calculating 20 lb Proportion (4)

<table>
<thead>
<tr>
<th></th>
<th>(Upto 2 decimal places)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate ( A \times 20\text{lb} ) -</td>
<td>19.00</td>
</tr>
<tr>
<td>Unburnt ( B \times 20\text{lb} ) -</td>
<td>0.94</td>
</tr>
<tr>
<td>Metal ( C \times 20\text{lb} ) -</td>
<td>3.96</td>
</tr>
<tr>
<td><strong>Total</strong> 20 lb</td>
<td></td>
</tr>
</tbody>
</table>

**Rounded Up/down (Lbs)**

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate - 19.0</td>
<td>Lb.</td>
</tr>
<tr>
<td>Unburnt - 0.5</td>
<td>Lb.</td>
</tr>
<tr>
<td>Metal - 4.0</td>
<td>Lb.</td>
</tr>
<tr>
<td><strong>Total</strong> 20.5 Lb</td>
<td></td>
</tr>
</tbody>
</table>
Hampton/NASA Steam Plant
Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0816–SST18

Sample Collection: Mon. Aug 8, 2016
QA Sample Preparation: Tue. Aug 9, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon on Mon. Aug 8, 2016 & will continue until 7:00 pm on Mon. Aug 8, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to following schedule/QA procedures:

**Mon. Aug 8:**

**AM Team**

Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition. Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

Starting:
12:00 noon

Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

Ending:
19:00 hrs

Continue 1-minute residue ash grab every hour as per above

Self-dumping ash sample hopper will be kept locked by OEO on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (Set these conditions before collecting samples)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>


**Tue. Aug 9**

07:30–10:00 AM  Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.

Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.

Thank you,

Anil Mehrotra, Plant Engineer

\[ \text{Fn 8/8/16 CTeam D. Woodan} \]

\[ \text{12 strokes/min} \rightarrow 125 \text{ ml} / \text{l} \]

\[ \text{12 strokes/min} = \frac{25 \text{ ml}}{\text{min}} \]
# REPORT OF ANALYSIS

**CLIENT:** Hampton/NASA Steam Plant  
**ATTN:** Anil Mehrotra, Plant Engineer  
**ADDRESS:** 50 Wythe Creek Road  
**CITY:** Hampton, VA 23666  
**PHONE:** (757) 805-1914  
**FAX:** e: amehrotra@hampton.gov  
**DATE:** 8/9/2016  
**TIME:** 1455  
**GRAB COLLECTION**  
**COLLECTED BY:** CLIENT  
**PICK UP BY:** CLIENT  
**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION:** ✓ Good  
**REPORT NO:** 16-14111  
**15:56**

**SAMPLE ID:** HSP-0816/SST18  
**SAMPLE NO:** 16-14111

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>TLG</th>
<th>08/12/16</th>
<th>1452</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt;0.050</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1452</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>100</td>
<td>0.562</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1452</td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.017</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1452</td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt;0.010</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1452</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt;0.050</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1452</td>
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<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
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<td>mg/L</td>
<td>TLG</td>
<td>08/11/16</td>
<td>1218</td>
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<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.050</td>
<td>1</td>
<td>&lt;0.050</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1452</td>
</tr>
<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt;0.010</td>
<td>mg/L</td>
<td>TLG</td>
<td>08/12/16</td>
<td>1452</td>
</tr>
</tbody>
</table>

---

Date: Aug. 9, 2016  
Sod. Sulphide Treatment Rea.  
Pump Setting: 12 strokes/min  
SST Rate: 9.5 gal. per day  
Results: Sat  
Reset Sod. Sulphide pump Setting  
To 12 strokes/min  

---

James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498  
VELAP# 460013  
EPA# VA00015
REPORT OF ANALYSIS

NOTES:
JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.
Reproduction of this report is not permitted, except in full, without written approval from James R. Reed & Associates.
These results on this report relate only to the sample(s) provided for analysis.
Results conform to NELAC standards, where applicable, unless otherwise indicated.
Initial pH: 10.87
Extraction Fluid used: 1
Final (end point) pH of Extraction Fluid: 6.67

Authorized By: 
Elaine Claiborne, Laboratory Director
Date: 12-Aug-16
HAMPTON/NASA STEAM PLANT
Residue ash Sample Data Records

<table>
<thead>
<tr>
<th>SAMPLE #</th>
<th>HSP-0316-5578</th>
<th>Sample Date:</th>
<th>Method Used:</th>
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<tbody>
<tr>
<td>Sample Certified Valid:</td>
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<table>
<thead>
<tr>
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<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
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<td>1795</td>
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<td>1930</td>
<td>2030</td>
</tr>
<tr>
<td>Initials</td>
<td>293/10</td>
<td>297/12</td>
<td>307/20</td>
<td>308/20</td>
<td>310/99</td>
<td>312/99</td>
<td>313/14</td>
<td>313/17</td>
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<th>10</th>
<th>11</th>
<th>12</th>
<th>13</th>
<th>14</th>
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<th>16</th>
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<table>
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<tr>
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<th>18</th>
<th>19</th>
<th>20</th>
<th>21</th>
<th>22</th>
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<tbody>
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<td></td>
</tr>
</tbody>
</table>

**WEIGHT TALLY SHEET (+ or - 1/2 Lb)**

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>40</td>
<td>15.</td>
</tr>
<tr>
<td>2.</td>
<td>38</td>
<td>16.</td>
</tr>
<tr>
<td>3.</td>
<td>39 1/2</td>
<td>17.</td>
</tr>
<tr>
<td>4.</td>
<td>40 1/2</td>
<td>18.</td>
</tr>
<tr>
<td>5.</td>
<td>38 1/2</td>
<td>19.</td>
</tr>
<tr>
<td>6.</td>
<td>41 1/2</td>
<td>20.</td>
</tr>
<tr>
<td>7.</td>
<td>38 1/2</td>
<td>21.</td>
</tr>
<tr>
<td>8.</td>
<td>40</td>
<td>22.</td>
</tr>
<tr>
<td>9.</td>
<td>38</td>
<td>23.</td>
</tr>
<tr>
<td>10.</td>
<td>43 1/2</td>
<td>24.</td>
</tr>
<tr>
<td>11.</td>
<td>42 1/2</td>
<td>25.</td>
</tr>
<tr>
<td>13.</td>
<td>45</td>
<td>27.</td>
</tr>
</tbody>
</table>

Unburnt Combustibles

1. 55
2. 48
3. 38
4. 43 1/2
5. 30 1/2
6. 

Unburnable (Metal)

1. 19
2. 18 1/2
3. 16
4. 8 1/2
5. 
6. 
Residue ash Sample Data Records
(Please Sign and Date below)

Sample #: HS0-0816 - SST18

<table>
<thead>
<tr>
<th>Wet Trench pH</th>
<th>Boilers Operating</th>
<th>Weather</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.2</td>
<td>142</td>
<td>Cloudy Rain</td>
</tr>
</tbody>
</table>

Chemical Treatment/Injection Rate: 12 m³ per minute 0.95 gpd (12 strokes/min).

Comments: MAGNET TURN OFF, METAL MIX WITH ASH.
DID MACHING MORNING OF 8/15 (it 8/8/16)

Team(s): A. Smith, P. Dothan, D. Terbo, J. Mercer

Quality Leader: Name: Dampier Womano          Designation: Op. Env

Team Leader: Name: Jakey Dyke                   Designation: Asst. Ho two

Mass Proportion Samples

<table>
<thead>
<tr>
<th></th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>642</td>
<td>69.86</td>
<td>13.97 = 14 lb</td>
<td>Total: 22.5 Lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>215</td>
<td>23.39</td>
<td>4.48 = 5 lb</td>
<td>With bucket and lid.</td>
</tr>
<tr>
<td>Unburnable</td>
<td>62</td>
<td>6.75</td>
<td>1.35 = 1 lb</td>
<td></td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: [Signature]
Date: 5/8/16
Calculation Sheet

20 lb. Ash Sample

Sample # HSP-0816-SS718

Date: 8/9/16

1. Sub Total (lbs)
   a. Aggregate 642
   b. Unburnt 215
   c. Metal 62
   d. TOTAL 919

2. Main Proportion (%)
   A. Aggregate \((\frac{a}{d} \times 100\%)\) 69.86
   B. Unburnt \((\frac{b}{d} \times 100\%)\) 23.39
   C. Metal \((\frac{c}{d} \times 100\%)\) 6.75
   D. 100%

3. Calculate 20 lb Proportion (lb)
   (Up to 2 decimal places)
   Aggregate \((A \times 20 lb)\) 1397
   Unburnt \((B \times 20 lb)\) 468
   Metal \((C \times 20 lb)\) 135
   Total 20 lb

4. Rounded Up-down (lbs)
   (To 1/2 lb)
   Aggregate 14 lb
   Unburnt 5.5 lb
   Metal 6 lb
   Total 20 lb
Hampton/NASA Steam Plant

Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0816–SST19-R3

Sample Collection: Tue. Aug 16, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon on Tue. Aug 16, 2016 & will continue until 7:00 pm on Tue. Aug 16, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to following schedule/QA procedures:

**Tue. Aug 16:**

**AM Team**

Ensure all test conditions given in Test Day Control Parameters below are set accordingly. **Mention specific existing condition.**

Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

**Starting:**
12:00 noon

Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.

**Ending:**
19:00 hrs:

Continue 1-minute residue ash grab every hour as per above

Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

<table>
<thead>
<tr>
<th>Test Day Control Parameters (Set these conditions before collecting samples)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reagent Specific Gravity</td>
</tr>
<tr>
<td>Fabric Filter Inlet Temp.</td>
</tr>
<tr>
<td>Boilers steam rate</td>
</tr>
<tr>
<td>Boilers running condition</td>
</tr>
</tbody>
</table>

**Remarks:**

* 1. Mechanical issues
* 2. Upset condition: trench cleaning/ wet trash
* 3. Ash conveyor issues
* 4. Others

**Wed. Aug 17**

07:30–10:00 AM Use Method HSP-3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.

Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.

Thank you.

Anil Mehrotra, Plant Engineer
Mehrotra, Anil

From: Paula Jackson <p.jackson@jrreedom.com>
Sent: Tuesday, September 20, 2016 10:15 AM
To: Mehrotra, Anil
Subject: TCLP re-extraction and comparison analysis

Anil,
You will be receiving two reports that summarize the re-extraction analysis that we recently performed on your TCLP samples.
The following is a more detailed explanation. Please call me if you have any questions.

The re-extraction/analysis from sample received 8/17/16 duplicated the original results. Based on initial chemistries, we were required to use extraction fluid #2. Analysis confirms the sample is hazardous for Pb. Cadmium was quantitated at 0.003mg/L (which is just under the hazardous limit).

However, the re-extraction/analysis from sample received 8/31/16 did not duplicate. Based on initial chemistries, we were required to use extraction fluid #2. This differs from the previous extraction of fluid #1. It is important to note the final endpoint pH differs dramatically for each extraction 8.29/5.18 respectively. This explains the dramatic difference in the quantitation of metals. Metals will leach readily in an acidic environment. Consequently, the re-extraction/analysis for this sample is hazardous for Pb.

Paula Jackson
Inorganic Department Supervisor
757-873-4703
## REPORT OF ANALYSIS

**CLIENT:** Hampton/NASA Steam Plant  
**ATTN:** Aml Mehrrota, Plant Engineer  
**ADDRESS:** 50 Wythe Creek Road  
**CITY:** Hampton, VA 23666  
**PHONE:** (757) 865-1914  
**FAX:** e: amehrota@hampton.gov

**SAMPLE RECEIPT**  
**DATE:** 8/1/2016  
**TIME:** 12:15  
**GRAB COLLECTION**  
**DATE:** 8/17/2016  
**TIME:** 00:00  
**COLLECTED BY:** CLIENT  
**PICK UP BY:** REED - AC  
**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION ☑️ Good | ☑️ Other (See C-O-C)**  
**REPORT NO:** 16-15518  
**14:12**

---

**SPECIAL NOTES:**

Supplement to Report No: 16-15518  
Revised Re-analysis (See Notes)

**SAMPLE ID:** HNSP-0816-SST19-A-Q3  
**SAMPLE NO:** 16-15518

---

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<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Date/Time</th>
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<td>D004</td>
<td>6010C</td>
<td>0.030</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
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<tr>
<td>Barium</td>
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<td>mg/L</td>
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<td>&lt; 0.010</td>
<td>mg/L</td>
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James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498  
VELAP# 460013  
EPA# VA00015

Page 1 of 2
REPORT OF ANALYSIS

NOTE:
JRA Quantitation Level is the concentration of the lowest calibration standard above zero with a reliable signal.

Reproduction of this report is not permitted, except in full, without written approval from James R. Reed & Associates.
The results on this report relate only to the sample(s) provided for analysis.

Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.30
Extraction Fluid used: #1
Final (end point) pH of Extraction Fluid: 8.29
Re-extracted 9/6/2016 ✓
Initial pH: 11.24
Extraction Fluid used: #2
Final (end point) pH of Extraction Fluid: 5.18
Sample hazardous for Lead - 5.64 mg/L,
Non Hazardous for Cadmium - 0.675 mg/L

RESPECTFULLY SUBMITTED

Elaine Chilborne
Laboratory Director
20-Sep-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax: (757) 873-1498
VELAP# 460013
EPA# VA00015

Page 2 of 2
### REPORT OF ANALYSIS

CLIENT: Hampton/NASA Steam Plant
ATTN: Anil Mehrotra, Plant Engineer
ADDRESS: 50 Wythe Creek Road
CITY: Hampton, VA 23666
PHONE: (757) 865-1914
FAX: e: amehrotra@hampton.gov

DATE: 8/17/2016  TIME: 0000
COLLECTED BY: CLIENT
PICK UP BY: REED - AC
NUMBER OF CONTAINERS: 1
GOOD CONDITION: ☑ Good  □ Other (See C-O-C)

REPORT NO: 16-15518 7:40

 SAMPLE ID: HNSP-0816-SST18-A-03
 SAMPLE NO  16-15518

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<th>Regulatory Leve</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
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<td>6010C</td>
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James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703  •  Fax: (757) 873-1498

VELAP# 460013
EPA# VA00015

Page 1 of 2
REPORT OF ANALYSIS

NOTE:

JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.

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The results on this report relate only to the sample(s) provided for analysis.

Results conform to NELAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.30
Extraction Fluid used: #1
Final (end point) pH of Extraction Fluid: 8.29

RESPECTFULLY SUBMITTED

Elaine Claborn
Laboratory Director
07-Sep-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax: (757) 873-1498

VELAP#: 460013
EPA#: VA00013

Page 2 of 2
ANALYSIS SAMPLE
CHAIN OF CUSTODY

SAMPLE NUMBER: HNG-0816-35719-A-63
MATERIAL: RFUSE

SAMPLE PREPARED BY: TONY TAYLOR
METHOD: HSP - 3A

16,15518

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Q.A. Weight: 22.5 lbs
Test: TCLP
All Metals

Reviewed By: [Signature]
Date: [Signature] - [Date]

Hampton/NASA Steam Plant
50 Wythe Creek Road, Hampton, Virginia (757) 865-6319 fax (757) 865-6217
# REPORT OF ANALYSIS

**CLIENT:** Hampton/NASA Steam Plant  
**ATTN:** Anil Mehrotra, Plant Engineer  
**ADDRESS:** 50 Wythe Creek Road  
**CITY:** Hampton, VA 23666  
**PHONE:** (757) 865-1914  
**FAX:** e: anil.mehrotra@hampton.gov  

**SAMPLE RECEIPT**  
**DATE:** 7/2016  
**TIME:** 14:15  
**GRAB COLLECTION**  
**DATE:** 8/17/2016  
**TIME:** 00:00  
**COLLECTED BY:** CLIENT  
**PICK UP BY:** REED - AC  
**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION:** ✔️ Good  

**SPECIAL NOTES:**  
Supplement to Report No. 16-14702  
Revised Re-analysis (See Notes)  
**REPORT NO:** 16-14702  
**14:12**  

**SAMPLE ID:** HNSP-0016-SST19-Q3  
**SAMPLE NO.** 16-14702

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<th>Result</th>
<th>Unit</th>
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<td>6010C</td>
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</table>

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*James R. Reed & Associates*

770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498  

Page 1 of 2
REPORT OF ANALYSIS

NOTE

RQA Quantitation Level is the concentration of the lowest calibration standard above zero with a reliable signal.

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The results on this report relate only to the sample(s) provided for analysis.

Results conform to NEJAC standards, where applicable, unless otherwise indicated.

Initial pH: 11.56
Extraction Fluid used: 2
Final (end point) pH of Extraction Fluid: 5.40

Re-extracted 9/8/2016
Initial pH: 11.10
Extraction Fluid used: 2
Final (end point) pH of Extraction Fluid: 5.43
Sample confirmed Hazardous for Lead - 5.64 mg/L,
Non Hazardous for Cadmium - 0.803 mg/L

RESPECTFULLY SUBMITTED

Elaine Chihoine
Laboratory Director
20-Sep-16

James R. Reed & Associates
770 Pilot House Drive, Newport News, VA 23606
(757) 873-4703 • Fax: (757) 873-1498

VELAP#: 460013
EPA#: VA00015

Page 2 of 2
# REPORT OF ANALYSIS

CLIENT: Hampton/NASA Steam Plant  
ATTN: Anil Mehrotra, Plant Engineer  
ADDRESS: 50 Wythe Creek Road  
CITY: Hampton, VA 23666  
PHONE: (757) 865-1914  
FAX: e.mehrotra@hampton.gov

SAMPLE RECEIVED  
DATE: 8/17/2016  
TIME: 1415  
GRAB COLLECTION  
DATE: 8/17/2016  
TIME: 0000  
COLLECTED BY: CLIENT  
PICK UP BY: REED - AC  
NUMBER OF CONTAINERS: 1  
GOOD CONDITION: ☑️ Good

SPECIAL NOTES: Retest Archive Sample

SAMPLE ID: HNSP-5816-SST19-Q3  
SAMPLE NO: 16-14702

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<th>Parameter</th>
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<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result (mg/L)</th>
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<td>Barium</td>
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<td>PEJ 8/30/2011</td>
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NOTE: *JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal.

REPRODUCTION OF THIS REPORT IS NOT PERMITTED, EXCEPT AS FURTHER APPROVED BY JAMES R. REED & ASSOCIATES.

Initial pH: 11.35  
Extraction Fluid used: 2  
Final (end point) pH of Extraction Fluid: 5.40

RESPECTFULLY SUBMITTED

Elaine Clahorne  
Laboratory Director  
30-Aug-16

Investigated by contracted test lab
James R. Reed. The lab is pulling samples from lot 2 batch 23192419

James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498

VELAP# 460013  
EPA# VA00015

Page 1 of 1
# ANALYSIS SAMPLE CHAIN OF CUSTODY

**17 Aug 16**

**SAMPLE NUMBER:** HNSP-0816-557198 & MATERIAL: Refuse

**SAMPLE PREPARED BY:** Tony Ford & METHOD: HSP-3A

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<td>0855</td>
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<tr>
<td>Austin</td>
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<td>Hurley</td>
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</tr>
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Q. A. Weight: 22.5 lbs

Test: TCLP

Reviewed By: [Signature]
Date: 9/8/16

Hampton/NASA Steam Plant
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

- **Sample #**: HSP-0816-55779-03
- **Sample Date**: 8/17/16
- **Method Used**: 7C1P-7A

**Sample Certified Valid:**

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**GRAB**

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### WEIGHT TALLY SHEET (+ or - 1/2Lb)

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<th>Aggregate</th>
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<td>2. 49.0</td>
<td>16.</td>
<td>2. 40.0</td>
</tr>
<tr>
<td>3. 48.5</td>
<td>17.</td>
<td>3.</td>
</tr>
<tr>
<td>4. 54.5</td>
<td>18.</td>
<td>4.</td>
</tr>
<tr>
<td>5. 54.0</td>
<td>19.</td>
<td>5. 7.5/4</td>
</tr>
<tr>
<td>6. 48.0</td>
<td>20.</td>
<td>6. 8.75/0</td>
</tr>
<tr>
<td>7. 49.0</td>
<td>21.</td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>8. 47.5</td>
<td>22.</td>
<td></td>
</tr>
<tr>
<td>9. 46.0</td>
<td>23.</td>
<td></td>
</tr>
<tr>
<td>10. 50.0</td>
<td>24.</td>
<td></td>
</tr>
<tr>
<td>11. 41.0</td>
<td>25.</td>
<td></td>
</tr>
<tr>
<td>12. 42.5</td>
<td>26.</td>
<td></td>
</tr>
<tr>
<td>13. 56.0</td>
<td>27.</td>
<td></td>
</tr>
<tr>
<td>14. 49.0</td>
<td>28.</td>
<td></td>
</tr>
<tr>
<td>15. 7.5</td>
<td>Unburnt Combustibles</td>
<td>1. 22.0</td>
</tr>
<tr>
<td>16.</td>
<td>2.</td>
<td></td>
</tr>
<tr>
<td>17.</td>
<td>3.</td>
<td></td>
</tr>
<tr>
<td>18.</td>
<td>4. Total = 22</td>
<td></td>
</tr>
<tr>
<td>19.</td>
<td>5. 2.82/6</td>
<td></td>
</tr>
<tr>
<td>20.</td>
<td>6.</td>
<td></td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample # HNSP-0816-55T19Q2

<table>
<thead>
<tr>
<th>Wet Trench pH: 8.2</th>
<th>Boilers Operating: 142</th>
<th>Weather: Sunny</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Treatment/Injection Rate: 12 Strokes/min</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Comments: Parameter for test: specific gravity 1011, CO control 600 ppm, FF temp 430°F with all 3 modules, Boiler steam rate 3314 kph, Sodium Sulfide rate 12 strokes per min.

Team(s): C4D

Quality Leader: Name Jack Jyp Designation: 

Team Leader: Name Tony Taylor Designation: 

Mass Proportion Samples

<table>
<thead>
<tr>
<th></th>
<th>Lbs</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>687.5</td>
<td>88.4</td>
<td>17.5</td>
<td>Total: 22.5 Lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>68</td>
<td>8.75</td>
<td>2.0</td>
<td>With bucket and lid.</td>
</tr>
<tr>
<td>Unburnable</td>
<td>22</td>
<td>2.82</td>
<td>1.5</td>
<td></td>
</tr>
</tbody>
</table>

TOTAL: 777.6

Verification by Quality Leader or Team Leader

Sign: [Signature]

Date: 8/17/16
Hampton/NASA Steam Plant
Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP–0816–SST20


An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at 12:00 noon on Wed. Aug 24, 2016 & will continue until 7:00 pm on Wed. Aug 24, 2016. The sample will be gathered, prepared, and sealed for analysis as per Method HSP–3A according to following schedule/QA procedures:

**Wed. Aug 24:**
- **AM Team**
  - Ensure all test conditions given in Test Day Control Parameters below are set accordingly. Mention specific existing condition.
  - Move the residue truck out from underneath the shaker pan and bring self-dumping sample collection hopper near shaker pan.

  **Starting:**
  - 12:00 noon

  **Operating Engineer on watch will randomly signal the residue handling operator to move the hopper underneath the pan and will let the residue ash drop directly in hopper for 1 minute.**

  **Ending:**
  - 19:00 hrs:
    - Continue 1-minute residue ash grab every hour as per above
    - Self-dumping ash sample hopper will be kept locked by OE on watch during entire ash collection period until ready for sorting.

| Test Day Control Parameters (Set these conditions before collecting samples) |
|-----------------------------|----------------|-----------------|
| Reagent Specific Gravity    | 1.01           | 5O<sub>2</sub> Control | 60 ppm |
| Fabric Filter Inlet Temp.   | 430°F          | FF Modules on   | All 3  |
| Boilers steam rate          | 33 kpph        | Sodium Sulfide rate | 12 Strokes/min |
| Boilers running condition   | Normal: Y/N *  | Any important change |

<table>
<thead>
<tr>
<th>Remarks:</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Mechanical issues</td>
</tr>
<tr>
<td>2. Upset condition: trench cleaning/ wet trash</td>
</tr>
<tr>
<td>3. Ash conveyor issues</td>
</tr>
<tr>
<td>4. Others</td>
</tr>
</tbody>
</table>

**Thu. Aug 25**

07:30–10:00 AM  Use Method HSP–3A for ash sorting and preparation of a 20 lb sample for laboratory analysis and another 20 lb archive sample.

Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.

Thank you,

Anil Mehrotra, Plant Engineer
### REPORT OF ANALYSIS

**CLIENT:** Hampton/NASA Steam Plant  
**ATTN:** Anil Mehrotra, Plant Engineer  
**ADDRESS:** 50 Wythe Creek Road  
**CITY:** Hampton, VA 23666  
**PHONE:** (757) 865-1914  
**FAX:** e: amehrotra@hampton.gov  
**SPECIAL NOTES:**

**DATE:** 8/21/2016  
**TIME:** 10:25  
**GRAB COLLECTION**  
**DATE:** 8/25/2016  
**TIME:** 00:00  
**COLLECTED BY:** CLIENT  
**PICK UP BY:** REED - BS  
**NUMBER OF CONTAINERS:** 1  
**GOOD CONDITION** ☑ Good  □ Other (See C-O-C)

---

**SAMPLE ID:** HSP-0816-SST20  
**SAMPLE NO:** 16-15218

<table>
<thead>
<tr>
<th>Parameter</th>
<th>EPA HW No.</th>
<th>Method Number</th>
<th>JRA QL</th>
<th>Regulatory Level</th>
<th>Result</th>
<th>Unit</th>
<th>Analyst/Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Toxic Characteristic</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Leaching Procedure</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>by SW-846 Method 1311</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Arsenic</td>
<td>D004</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>PEJ 8/30/2011708</td>
</tr>
<tr>
<td>Barium</td>
<td>D005</td>
<td>6010C</td>
<td>0.250</td>
<td>103</td>
<td>0.555</td>
<td>mg/L</td>
<td>PEJ 8/30/2011708</td>
</tr>
<tr>
<td>Cadmium</td>
<td>D006</td>
<td>6010C</td>
<td>0.005</td>
<td>1</td>
<td>0.315</td>
<td>mg/L</td>
<td>PEJ 8/30/2011708</td>
</tr>
<tr>
<td>Chromium</td>
<td>D007</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 8/30/2011708</td>
</tr>
<tr>
<td>Lead</td>
<td>D008</td>
<td>6010C</td>
<td>0.050</td>
<td>5</td>
<td>0.102</td>
<td>mg/L</td>
<td>PEJ 8/30/2011708</td>
</tr>
<tr>
<td>Mercury</td>
<td>D009</td>
<td>7470A</td>
<td>0.0002</td>
<td>2</td>
<td>&lt; 0.0002</td>
<td>mg/L</td>
<td>TLG 8/30/2011651</td>
</tr>
<tr>
<td>Selenium</td>
<td>D010</td>
<td>6010C</td>
<td>0.050</td>
<td>1</td>
<td>0.202</td>
<td>mg/L</td>
<td>PEJ 8/30/2011708</td>
</tr>
<tr>
<td>Silver</td>
<td>D011</td>
<td>6010C</td>
<td>0.010</td>
<td>5</td>
<td>&lt; 0.010</td>
<td>mg/L</td>
<td>PEJ 8/30/2011708</td>
</tr>
</tbody>
</table>

*NOTE:* JRA Quantification Level is the concentration of the lowest calibration standard above zero with a reliable signal. Reproduction of this report is not permitted, except in full, without written approval from James R. Reed & Associates.

**Initial pH:** 11.37  
**Extraction Fluid used:** 1  
**Final (end point) pH of Extraction Fluid:** 7.88

---

**RESPECTFULLY SUBMITTED**

Elaine Claiborne  
Laboratory Director  
30-Aug-16

---

James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498  
VELAP# 460013  
EPA# VA00015

Page 1 of 1
# ANALYSIS SAMPLE
## CHAIN OF CUSTODY

**SAMPLE NUMBER:** HSP-0516-SST20  
**MATERIAL:** Residual Ash Data  
**SAMPLE PREPARED BY:** Dwayne Landers  
**METHOD:** HSP-3A

<table>
<thead>
<tr>
<th>CUSTODIAN</th>
<th>FROM</th>
<th>TO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dwayne Landers</td>
<td>5/25/16</td>
<td>07/00</td>
</tr>
<tr>
<td></td>
<td>8/25/16</td>
<td>08/04</td>
</tr>
<tr>
<td></td>
<td>9/25/16</td>
<td>09/08</td>
</tr>
<tr>
<td></td>
<td>9/25/16</td>
<td>10/25</td>
</tr>
</tbody>
</table>

**Q. A. Weight:** 22.5 lbs  
**Test:** TLC P - All Metals

Reviewed By:  
Date: 8/30

Hampton/NASA Steam Plant  
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

**SAMPLE #**: HPS-0816-SS-T20  
**Sample Date**: 8/12/77  
**Method Used**: HSP-3A

<table>
<thead>
<tr>
<th>GRAB</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>0958</td>
<td>1106</td>
<td>1050</td>
<td>1306</td>
<td>1408</td>
<td>1516</td>
<td>1650</td>
<td>1748</td>
</tr>
<tr>
<td>Initials</td>
<td>749</td>
<td>702</td>
<td>749</td>
<td>702/PC</td>
<td>702/PC</td>
<td>702</td>
<td>702</td>
<td>702</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>GRAB</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
<th>13</th>
<th>14</th>
<th>15</th>
<th>16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initials</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>GRAB</th>
<th>17</th>
<th>18</th>
<th>19</th>
<th>20</th>
<th>21</th>
<th>22</th>
<th>23</th>
<th>24</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Initials</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### WEIGHT TALLY SHEET (+ or - ½ Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 41</td>
<td>15.</td>
<td>Unburnt Combustibles</td>
</tr>
<tr>
<td>2. 54.5</td>
<td>16.</td>
<td>1. 34.5</td>
</tr>
<tr>
<td>3. 51.5</td>
<td>17.</td>
<td>2. 53</td>
</tr>
<tr>
<td>4. 44</td>
<td>18.</td>
<td>3. 31</td>
</tr>
<tr>
<td>5. 49</td>
<td>19.</td>
<td>4.</td>
</tr>
<tr>
<td>6. 58.5</td>
<td>20.</td>
<td>5.</td>
</tr>
<tr>
<td>7. 45.5</td>
<td>21.</td>
<td>6.</td>
</tr>
<tr>
<td>8. 38</td>
<td>22.</td>
<td>Unburnable (Metal)</td>
</tr>
<tr>
<td>9. 2.0</td>
<td>23.</td>
<td>1. 13.5</td>
</tr>
<tr>
<td>10. 43</td>
<td>24.</td>
<td>2.</td>
</tr>
<tr>
<td>11. 43.5</td>
<td>25.</td>
<td>3.</td>
</tr>
<tr>
<td>12. 73</td>
<td>26.</td>
<td>4.</td>
</tr>
<tr>
<td>13.</td>
<td>27.</td>
<td>5.</td>
</tr>
<tr>
<td>14.</td>
<td>28.</td>
<td>6.</td>
</tr>
</tbody>
</table>
Residue ash Sample Data Records
(Please Sign and Date below)

Sample # HSP-08/14-SST20

<table>
<thead>
<tr>
<th>Wet Trench pH: 8.6</th>
<th>Boilers Operating: 1+2*</th>
<th>Weather: <strong>Clear &amp; Sunny</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Treatment/ Injection Rate: SST @ 12 strokes per minute</td>
<td>45 gallons per day</td>
<td></td>
</tr>
<tr>
<td>Comments: <em>Closed door on #1 blr due to tube leak at 0850. #1 blr came off line at 1015.</em>*</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Team(s): D. Woodward, J. Taylor, J. Jones, R. Late

Quality Leader: Name D. Woodward Designation: OP Line

Team Leader: Name Jackie Davis Designation: Ash Handler

### Mass Proportion Samples

<table>
<thead>
<tr>
<th></th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>555.5</td>
<td>80.8</td>
<td>16</td>
<td>Total: 22.5 Lbs.</td>
</tr>
<tr>
<td>Unburnt</td>
<td>118.5</td>
<td>17.24</td>
<td>3.5</td>
<td>With bucket and lid.</td>
</tr>
<tr>
<td>Unburnable</td>
<td>13.5</td>
<td>1.96</td>
<td>.5</td>
<td></td>
</tr>
</tbody>
</table>

Verification by Quality Leader or Team Leader

Sign: [Signature]

Date: 8/25/10
### Calculation Sheet

**20 lb. Ash Sample**

**Sample #:** HSP-0516-SS72D  
**Date:** 8/25/16

<table>
<thead>
<tr>
<th>Sub Total (Lbs)</th>
<th>Mass Proportion (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>a. Aggregate</strong></td>
<td><strong>A. Aggregate (a/d x 100%)</strong></td>
</tr>
<tr>
<td>666.5</td>
<td>80.8</td>
</tr>
<tr>
<td><strong>b. Unburnt</strong></td>
<td><strong>B. Unburnt (b/d x 100%)</strong></td>
</tr>
<tr>
<td>118.5</td>
<td>17.24</td>
</tr>
<tr>
<td><strong>c. Metal</strong></td>
<td><strong>C. Metal (c/d x 100%)</strong></td>
</tr>
<tr>
<td>13.5</td>
<td>1.96</td>
</tr>
<tr>
<td><strong>d. Total</strong></td>
<td><strong>D. Total 100%</strong></td>
</tr>
<tr>
<td>808.5</td>
<td></td>
</tr>
</tbody>
</table>

#### 3) Calculate 20 lb Proportion (Lb)

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate (A x 20lb)</td>
<td>16.16</td>
</tr>
<tr>
<td>Unburnt (B x 20lb)</td>
<td>3.45</td>
</tr>
<tr>
<td>Metal (C x 20lb)</td>
<td>0.29</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>20 lb</strong></td>
</tr>
</tbody>
</table>

#### 4) Rounded Up/Down (Lbs)

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>16.0 lb</td>
</tr>
<tr>
<td>Unburnt</td>
<td>3.5 lb</td>
</tr>
<tr>
<td>Metal</td>
<td>0.5 lb</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>20.0 lb</strong></td>
</tr>
</tbody>
</table>
Hampton/NASA Steam Plant
Procedure for Characterization of Residue Ash treatment with Sodium Sulfide

Sample Number: HNSP-0816–SST21
(During start-up of Boiler #1)
Sample Collection: Fri. Aug 26, 2016

An 8-hour residue ash sample collection with Sodium Sulfide injection is to start at
6:00 pm on Fri. Aug 26, 2016 & will continue until 1:00 am on Sat. Aug 27, 2016.
The sample will be gathered, prepared, and sealed for analysis as per Method HSP–
3A according to following schedule/QA procedures:

Fri. Aug 26:
AM Team
Ensure all test conditions given in Test Day Control Parameters
below are set accordingly. Mention specific existing condition.
Move the residue truck out from underneath the shaker pan and
bring self-dumping sample collection hopper near shaker pan.

Starting:
6:00 pm
Operating Engineer on watch will randomly signal the residue
handling operator to move the hopper underneath the pan and
will let the residue ash drop directly in hopper for 1 minute.

Ending:
01:00 am:
Self-dumping ash sample hopper will be kept locked by OE on
watch during entire ash collection period until ready for sorting.

Test Day Control Parameters (Set these conditions before collecting samples)

<table>
<thead>
<tr>
<th>Reagent Specific Gravity</th>
<th>1.01</th>
<th>SO₂ Control</th>
<th>60 ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fabric Filter Inlet Temp.</td>
<td>430°F</td>
<td>FF Modules on</td>
<td>All 3</td>
</tr>
<tr>
<td>Boilers steam rate</td>
<td>33 kpph</td>
<td>Sodium Sulfide rate</td>
<td>12 Strokes/min</td>
</tr>
<tr>
<td>Boilers running condition</td>
<td>Normal: Y/N</td>
<td>Any important change</td>
<td></td>
</tr>
</tbody>
</table>

3. Ash conveyor issues 4. Others

Fri. Sept 2:
07:30–10:00 AM Use Method HSP-3A for ash sorting and preparation of a 20 lb
sample for laboratory analysis and another 20 lb archive sample.

Always follow QA/QC and Chain of Custody procedures as per DEQ Permit # 297.

Thank you,
Anil Mehrotra, Plant Engineer
# REPORT OF ANALYSIS

**CLIENT:** Hampton/NASA Steam Plant  
**ATTN:** Anil Mehrotra, Plant Engineer  
**ADDRESS:** 50 Wythe Creek Road  
**CITY:** Hampton, VA 23666  
**PHONE:** (757) 865-1914  
**FAX:** anil.mehrotra@hampton.gov

**SAMPLE ID:** HSP-0816-SST21  
**SAMPLE NO:** 16-15353

---

**Parameter** | **EPA HW No.** | **Method Number** | **JRA QL** | **Regulatory Level** | **Result** | **Unit** | **Analyst/Date/Time**
---|---|---|---|---|---|---|---
Toxic Characteristic Leaching Procedure by SW-846 Method 1311  
**Arsenic** | D304 | 6010C | 0.050 | 5 | < 0.050 | mg/L | FEJ 9/6/2016 1321

**Barium** | D305 | 6010C | 0.250 | 100 | 0.369 | mg/L | FEJ 9/6/2016 1321

**Cadmium** | D306 | 6010C | 0.005 | 1 | 0.041 | mg/L | FEJ 9/6/2016 1321

**Chromium** | D307 | 6010C | 0.010 | 5 | < 0.010 | mg/L | FEJ 9/6/2016 1321

**Lead** | D308 | 6010C | 0.050 | 5 | < 0.050 | mg/L | FEJ 9/6/2016 1321

**Mercury** | D309 | 7470A | 0.0002 | 0.2 | < 0.0002 | mg/L | TLG 9/6/2016 1526

**Selenium** | D310 | 6010C | 0.050 | 1 | < 0.050 | mg/L | FEJ 9/6/2016 1321

**Silver** | D311 | 6010C | 0.010 | 5 | < 0.010 | mg/L | FEJ 9/6/2016 1321

---

James R. Reed & Associates  
770 Pilot House Drive, Newport News, VA 23606  
(757) 873-4703 • Fax: (757) 873-1498

VELAP# 460013  
EPA# VA00015

Page 1 of 2
Initial pH: 16.99
Extraction Fluid used: #1
Final (end point) pH of Extraction Fluid: 1.42

RESPECTFULLY SUBMITTED

Elaine Claiborne
Laboratory Director
07-Sep-16
## ANALYSIS SAMPLE CHAIN OF CUSTODY

**Sample Number:** HSP-0816-5731

**Material:** Res 109AC

**Sample Prepared By:** [Signature]

**Method:** HSP-3A

<table>
<thead>
<tr>
<th>Custodian</th>
<th>From</th>
<th>To</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>1000</td>
<td>28 Aug 16</td>
</tr>
<tr>
<td></td>
<td>1100</td>
<td>29 Aug 16</td>
</tr>
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**Q.A. Weight:** 22.5 LBS

**Test:** TCLP All Metals

Reviewed By: [Signature]

Date: 9/17

Hampton/NASA Steam Plant
50 Wythe Creek Road, Hampton, Virginia, (757) 865-1914 fax (757) 865-1317
### Hampton/NASA Steam Plant
Residue ash Sample Data Records

**Sample #**: HSP-08/16-55T21  
**Sample Date**: 26 Apr 16  
**Method Used**: HSP 3A  
**Sample Certified Valid**: [Handwritten annotation]

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<th>4</th>
<th>5</th>
<th>6</th>
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<td>19:05</td>
<td>20:07</td>
<td>21:09</td>
<td>22:03</td>
<td>23:02</td>
<td>00:00</td>
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</tbody>
</table>

### WEIGHT TALLY SHEET (+ or - 1/4 Lb)

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Aggregate</th>
<th>Other Materials Over 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 41.5</td>
<td>2. 42</td>
<td>Unburnt Combustibles 1. 36</td>
</tr>
<tr>
<td>3. 48</td>
<td>4. 43</td>
<td>2. 50.5</td>
</tr>
<tr>
<td>5. 47</td>
<td>6. 49</td>
<td></td>
</tr>
<tr>
<td>7. 49</td>
<td>8. 46</td>
<td></td>
</tr>
<tr>
<td>9. 45</td>
<td>10. 47</td>
<td>Unburnable (Metal) 1. 31.5</td>
</tr>
<tr>
<td>11. 47.5</td>
<td>12. 54</td>
<td></td>
</tr>
<tr>
<td>13. 49.5</td>
<td>14. 42</td>
<td></td>
</tr>
</tbody>
</table>

[Handwritten annotations]
Residue ash Sample Data Records
(Please Sign and Date below)

Sample #

<table>
<thead>
<tr>
<th>Wet Trench pH: 8.6</th>
<th>Boilers Operating: 1 &amp; 2</th>
<th>Weather: Muggy/Humid</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chemical Treatment/Injection Rate:</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Comments: Blows up Boiler/Light-off

Teams: ____________________________________________________________

Quality Leader: Name ___________________ Designation: ________________

Team Leader: Name JACK DYE Designation: ________________

Mass Proportion Samples

<table>
<thead>
<tr>
<th></th>
<th>Lbs.</th>
<th>%</th>
<th>20 Lb. Sample</th>
<th>Q.A. Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate</td>
<td>687</td>
<td>.9</td>
<td>18</td>
<td></td>
</tr>
<tr>
<td>Unburnt</td>
<td>87</td>
<td>.1</td>
<td>1.5</td>
<td></td>
</tr>
<tr>
<td>Unburnable</td>
<td>32</td>
<td>.04</td>
<td>.5</td>
<td></td>
</tr>
</tbody>
</table>

Total: 22.5 Lbs.
With bucket and lid.

Verification by Quality Leader or Team Leader

Sign: Jack Dye
Date: 28 Aug 16
Calculation Sheet

20 lb. Ash Sample

Sample # _ HNSP-0816-55121_

Date: 8/26/16

1. Subtotal (Lbs)
   a. Aggregate 6.89
   b. Unburnt 8.7
   c. Metal 3.2
   d. Total 8.07

2. Mass Proportion (%)
   A. Aggregate \( \frac{a}{A} \times 100\% \) = 9 \( (85\% \)
   B. Unburnt \( \frac{b}{A} \times 100\% \) = 1 \( (11\% \)
   C. Metal \( \frac{c}{A} \times 100\% \) = 0.4 \( (4\% \)
   D. Total 100%

3. Calculate 20 lb Proportion (Lbs)
   Aggregate \( a \times 20\text{lb} \) = 18 \( (17\) Lbs
   Unburnt \( b \times 20\text{lb} \) = 2 \( (2\) Lbs
   Metal \( c \times 20\text{lb} \) = 0.8 \( (0.8\) Lbs
   Total 20 Lbs

4. Rounded Up/Down (Ubs)
   Aggregate 18 \( 1/2 \) Lbs
   Unburnt 1.5 \( 1\) Lbs
   Metal 1.5 \( 1\) Lbs
   Total 20.0 Lbs
APPENDIX F

OTHER MSW PLANTS HEAVY METALS TCLP RESULTS

1. Covanta, Fairfax, Virginia

2. Wheelabrator, Portsmouth, Virginia
August 19, 2013

Mr. Richard Doucette  
Land Protection and Revitalization Program Manager  
Virginia Department of Environmental Quality  
Northern Virginia Regional Office  
13901 Crown Court  
Woodbridge, VA 22193-1453

Subject: Covanta Fairfax, Inc. Permit-by-Rule #545  
1st 2013 Semi-Annual TCLP Ash Sampling and Test Report

Dear Mr. Doucette,

Pursuant to PBR #545, Attachment II, Ash Residue Testing the 1st 2013 Semi-Annual TCLP Ash Sampling and Test Report is hereby submitted to demonstrate compliance:

In accordance with 9 VAC 20-80-370 D.8., the facility shall perform a chemical analysis of all residual ash generated in accordance with the facility's permit. Based on a statistical analysis performed using ash testing data from 1994 to 2009, and the quantity of ash generated by the facility, the facility shall perform semi-annual ash sampling and report results within 90 days of sample collection in accordance with 9 VAC 20-80-370 D.8.a.

The analytical data was evaluated in complete compliance with the procedures set forth and required by SW-846. The statistical evaluation has determined that the waste does not exhibit a hazardous characteristic and that it should be managed as a nonhazardous solid waste.

Please contact me if you have any questions or comments.

Regards,

Scott P. Drew  
Facility Manager

Cc: C. Forbes (Fairfax County Solid Waste)  
    L. Boone (Fairfax County Solid Waste)  
    D. Shabat (Osirka and Bartilucci)  
    J. Hermann (CBI)

Encls
### TABLE 3 LABORATORY RESULTS FOR THE COVANTA FAIRFAX, INC

#### 3.1 SAMPLE SPECIFIC RESULTS

<table>
<thead>
<tr>
<th>SAMPLE NUMBER</th>
<th>SAMPLE DATE</th>
<th>As (mg/l)</th>
<th>Be (mg/l)</th>
<th>Cd (mg/l)</th>
<th>Cr (mg/l)</th>
<th>Cu (mg/l)</th>
<th>Fe (mg/l)</th>
<th>Mn (mg/l)</th>
<th>Ni (mg/l)</th>
<th>Pb (mg/l)</th>
<th>Zn (mg/l)</th>
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<tr>
<td>FFX/CA/TCLP/13.05.28/1A</td>
<td>5/28/2013</td>
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<td>&lt;0.250</td>
<td>&lt;0.0500</td>
<td>&lt;0.250</td>
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#### 3.2 STATISTICAL RESULTS

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<td>DEGREES OF FREEDOM</td>
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<tr>
<td>SAMPLE MEAN (XBAR)</td>
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<td>0.250</td>
<td>0.250</td>
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<td>SAMPLE VARIANCE (S^2)</td>
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<td>0.02</td>
<td>0.000</td>
<td>0.06E+00</td>
<td>0.000</td>
<td>2.9E-07</td>
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<td>0.06E+00</td>
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<tr>
<td>STANDARD DEVIATION (S)</td>
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<td>0.000</td>
<td>0.06E+00</td>
<td>0.000</td>
<td>5.4E-04</td>
<td>0.06E+00</td>
<td>0.06E+00</td>
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<td>STD ERROR (S XBAR)</td>
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<td>0.04</td>
<td>0.000</td>
<td>0.06E+00</td>
<td>0.000</td>
<td>1.4E-04</td>
<td>0.06E+00</td>
<td>0.06E+00</td>
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<tr>
<td>80% CI Upper Limit (actual)</td>
<td>0.250</td>
<td>0.52</td>
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<td>0.00088</td>
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<td>0.250</td>
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<tr>
<td>80% CI Upper Limit (exp. of lognormal)</td>
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<td>0.64</td>
<td>0.050</td>
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<td>0.250</td>
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<td>0.050</td>
<td>0.250</td>
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<td>MINIMUM</td>
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<td>0.250</td>
<td>0.00010</td>
<td>0.050</td>
<td>0.250</td>
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#### 3.3 REGULATORY THRESHOLD

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<th>1.0</th>
<th>5.0</th>
<th>5.0</th>
<th>0.2</th>
<th>1.0</th>
<th>5.0</th>
</tr>
</thead>
</table>
### Table 4

**COMPARISON OF SW-846 STATISTICAL RESULTS AND REGULATORY THRESHOLDS FOR METAL ANALYTES**

<table>
<thead>
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<th>Analyte</th>
<th>90% Upper Confidence Interval per SW-846 (b)</th>
<th>Regulatory Threshold (a)</th>
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<tr>
<td>Metals</td>
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<td></td>
</tr>
<tr>
<td>Arsenic</td>
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<td>5.0</td>
</tr>
<tr>
<td>Barium</td>
<td>0.520</td>
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<td>Cadmium</td>
<td>0.050</td>
<td>1.0</td>
</tr>
<tr>
<td>Chromium</td>
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<td>5.0</td>
</tr>
<tr>
<td>Lead</td>
<td>0.250</td>
<td>5.0</td>
</tr>
<tr>
<td>Mercury</td>
<td>0.00088</td>
<td>0.2</td>
</tr>
<tr>
<td>Selenium</td>
<td>0.050</td>
<td>1.0</td>
</tr>
<tr>
<td>Silver</td>
<td>0.250</td>
<td>5.0</td>
</tr>
</tbody>
</table>

(a) 40 CFR Part 261. All units are expressed as milligrams per liter (mg/L).

(b) 90% Upper Confidence Interval as a single-tailed distribution is equivalent to an 80% Upper Confidence Interval as a two-tailed distribution.
March 23, 2016

Mr. Richard Doucette
Solid Waste Manager
Virginia Department of Environmental Quality
Northern Virginia Regional Office
13901 Crown Court
Woodbridge, VA 22193

Reference: Covanta Alexandria/Arlington, Inc. (CAAI) PBR #551
2016 Semi-Annual TCLP Ash Analysis

Dear Mr. Doucette,

CAAI has enclosed the first semi-annual TCLP Ash Test Report for 2016. The samples were collected from February 16-25, 2016.

Please contact me if you have any questions or comments.

Regards,

Bryan Donnelly
Facility Manager

BD/KMM

CC:
W. Skrabak (City of Alexandria)
K. Perrin (HDR)*
K. Perzyk (VADEQ)*

*Report sent via email.
COVANTA ENERGY GROUP, INC.
CEMS/TESTING DEPARTMENT

ENVIRONMENTAL TEST REPORT
FOR
COVANTA ALEXANDRIA/ARLINGTON, INC.

CEG REPORT NO.: 4093

REPORT DATE: March 23, 2016

PREPARED FOR: Alexandria Resource Recovery Facility

PURPOSE: Characterization of Ash Residue

SAMPLE PERIOD: February 16 through February 25, 2016

PREPARED BY: Covanta Energy Group, Inc.
Department 14 - CEM/Emission Testing
### TABLE 3 LABORATORY RESULTS FOR THE COVANTA ALEXANDRIA WARENKILM, INC

#### 3.1 SAMPLE SPECIFIC RESULTS

<table>
<thead>
<tr>
<th>SAMPLE ID</th>
<th>Cu (mg/L)</th>
<th>Be (mg/L)</th>
<th>As (mg/L)</th>
<th>Pb (mg/L)</th>
<th>Hg (mg/L)</th>
<th>Se (mg/L)</th>
<th>Ag (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alex/CA/CLP/16.02.16/1B</td>
<td>&lt; 0.250</td>
<td>&lt; 0.050</td>
<td>&lt; 0.250</td>
<td>&lt; 0.250</td>
<td>&lt; 0.00020</td>
<td>&lt; 0.050</td>
<td>&lt; 0.2500</td>
</tr>
<tr>
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<td>&lt; 0.050</td>
<td>&lt; 0.250</td>
<td>&lt; 0.250</td>
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</tr>
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</tr>
<tr>
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<td>&lt; 0.050</td>
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<td>&lt; 0.050</td>
<td>&lt; 0.250</td>
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<td>&lt; 0.00020</td>
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<td>&lt; 0.050</td>
<td>&lt; 0.250</td>
<td>&lt; 0.250</td>
<td>&lt; 0.00020</td>
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</tr>
<tr>
<td>Alex/CA/CLP/16.02.23/1A</td>
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<td>&lt; 0.050</td>
<td>&lt; 0.250</td>
<td>&lt; 0.250</td>
<td>&lt; 0.00020</td>
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<td>&lt; 0.2500</td>
</tr>
<tr>
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<td>&lt; 0.050</td>
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<td>&lt; 0.250</td>
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<td>&lt; 0.050</td>
<td>&lt; 0.2500</td>
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<td>&lt; 0.050</td>
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#### 3.2 STATISTICAL RESULTS

<table>
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<th>14</th>
<th>14</th>
<th>14</th>
<th>14</th>
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<th>14</th>
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<td>13</td>
<td>13</td>
<td>13</td>
<td>13</td>
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<tr>
<td>SAMPLE MEAN (XBAR)</td>
<td>0.250</td>
<td>0.97</td>
<td>0.030</td>
<td>0.250</td>
<td>0.250</td>
<td>0.00020</td>
<td>0.050</td>
<td>0.250</td>
</tr>
<tr>
<td>SAMPLE VARIANCE (SD2)</td>
<td>0.0E+00</td>
<td>0.01</td>
<td>0.000</td>
<td>0.0E+00</td>
<td>0.000</td>
<td>3.2E-39</td>
<td>0.0E+00</td>
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<tr>
<td>STANDARD DEVIATION (S)</td>
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<td>0.12</td>
<td>0.000</td>
<td>0.0E+00</td>
<td>0.000</td>
<td>5.0E-20</td>
<td>0.0E+00</td>
<td>0.0E+00</td>
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<tr>
<td>STD ERROR (S/XBAR)</td>
<td>0.0E+00</td>
<td>0.03</td>
<td>0.000</td>
<td>0.0E+00</td>
<td>0.000</td>
<td>1.5E-20</td>
<td>0.0E+00</td>
<td>0.0E+00</td>
</tr>
<tr>
<td>90% CI Upper Limit (actual)</td>
<td>0.260</td>
<td>1.919</td>
<td>0.050</td>
<td>0.260</td>
<td>0.260</td>
<td>0.00020</td>
<td>0.050</td>
<td>0.250</td>
</tr>
<tr>
<td>90% CI Upper Limit (esp. lognormal)</td>
<td>0.250</td>
<td>1.20</td>
<td>0.050</td>
<td>0.250</td>
<td>0.250</td>
<td>0.00020</td>
<td>0.050</td>
<td>0.250</td>
</tr>
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<td>MAXIMUM</td>
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<td>0.82</td>
<td>0.050</td>
<td>0.250</td>
<td>0.250</td>
<td>0.00020</td>
<td>0.050</td>
<td>0.250</td>
</tr>
<tr>
<td>MINIMUM</td>
<td>0.250</td>
<td>1.00</td>
<td>0.050</td>
<td>0.250</td>
<td>0.250</td>
<td>0.00020</td>
<td>0.050</td>
<td>0.250</td>
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#### 3.3 REGULATORY THRESHOLD

<table>
<thead>
<tr>
<th></th>
<th>Cu</th>
<th>Be</th>
<th>As</th>
<th>Pb</th>
<th>Hg</th>
<th>Se</th>
<th>Ag</th>
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<tbody>
<tr>
<td>Maximum</td>
<td>5.0</td>
<td>100</td>
<td>1.0</td>
<td>5.0</td>
<td>5.0</td>
<td>0.2</td>
<td>1.0</td>
</tr>
</tbody>
</table>

(a) Less than symbol (*) indicates laboratory result below the detection limit.
The value used in this table is one-half (1/2) of the detection limit provided by the laboratory.
### Table 4

COMPARISON OF SW-846 STATISTICAL RESULTS AND REGULATORY THRESHOLDS FOR METAL ANALYTES

<table>
<thead>
<tr>
<th>Analyte</th>
<th>90% Upper Confidence Interval per SW-846 (b)</th>
<th>Regulatory Threshold (a)</th>
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<tr>
<td>Metals</td>
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<td></td>
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<tr>
<td>Arsenic</td>
<td>0.250</td>
<td>5.0</td>
</tr>
<tr>
<td>Barium</td>
<td>1.012</td>
<td>100.0</td>
</tr>
<tr>
<td>Cadmium</td>
<td>0.050</td>
<td>1.0</td>
</tr>
<tr>
<td>Chromium</td>
<td>0.250</td>
<td>5.0</td>
</tr>
<tr>
<td>Lead</td>
<td>0.250</td>
<td>5.0</td>
</tr>
<tr>
<td>Mercury</td>
<td>0.002</td>
<td>0.2</td>
</tr>
<tr>
<td>Selenium</td>
<td>0.050</td>
<td>1.0</td>
</tr>
<tr>
<td>Silver</td>
<td>0.250</td>
<td>5.0</td>
</tr>
</tbody>
</table>

(a) 40 CFR Part 201. All units are expressed as milligrams per liter (mg/L).
(b) 90% Upper Confidence Interval as a single-tailed distribution is equivalent to an 80% Upper Confidence Interval as a two-tailed distribution.
April 9, 2015

Jeff Daibler
Waste Compliance Technical Coordinator
Virginia Department of Environmental Quality
5635 Southern Blvd.
Virginia Beach, VA 23462

Re: Wheelabrator Portsmouth WTE Ash Test Results

Dear Mr. Daibler,

In accordance with 9VAC20-81-340.E.7 please find enclosed a copy of the chemical analyses of residue Completed in February 2015.

If you have any questions about this submittal, you can reach me at 393-3105.

Sincerely,

Jeff Landrum
Environmental Manager

Enclosure
DOCUMENT CERTIFICATION

Facility Name: Wheelabrator Portsmouth Inc.
Registration No: 61018
Facility Location: Portsmouth, VA
Type of Submittal Attached: 2015 Residue Chemical Analysis Results

Certification: I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering and evaluating the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

Name of Responsible Official (Print): Rob Johnson
Title: Plant Manager
Signature: [Signature] Date: 04/09/2015
<table>
<thead>
<tr>
<th>SAMPLE ID</th>
<th>SAMPLED BY</th>
<th>DATE</th>
<th>AM/PM</th>
<th>As (mg/L)</th>
<th>Fe (mg/L)</th>
<th>Cu (mg/L)</th>
<th>Zn (mg/L)</th>
<th>Pb (mg/L)</th>
<th>Hg (mg/L)</th>
<th>Se (mg/L)</th>
<th>Ag (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6014</td>
<td>JL / JL</td>
<td>2/15/2015</td>
<td>AM</td>
<td>&lt; 0.2</td>
<td>&lt; 0.1</td>
<td>&lt; 0.01</td>
<td>&lt; 0.02</td>
<td>3</td>
<td>&lt; 0.0002</td>
<td>&lt; 0.2</td>
<td>&lt; 0.02</td>
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<td>JL / JL</td>
<td>2/19/2015</td>
<td>PM</td>
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<td>&lt; 0.01</td>
<td>&lt; 0.02</td>
<td>&lt; (14.2)</td>
<td>&lt; 0.0002</td>
<td>&lt; 0.2</td>
<td>&lt; 0.02</td>
<td></td>
</tr>
<tr>
<td>6016</td>
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<td>2/16/2015</td>
<td>AM</td>
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<td>0.6</td>
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<td>&lt; 0.02</td>
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<td>0.00031</td>
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<td>&lt; 0.02</td>
</tr>
<tr>
<td>6020</td>
<td>JL / JL</td>
<td>2/16/2015</td>
<td>PM</td>
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<td>&lt; 0.02</td>
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<tr>
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<td>AM</td>
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<td>1.0</td>
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<td>&lt; 0.02</td>
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<td>&lt; 0.02</td>
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<td>6022</td>
<td>MK / MK</td>
<td>2/17/2015</td>
<td>PM</td>
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<td>0.9</td>
<td>&lt; 0.01</td>
<td>&lt; 0.02</td>
<td>1</td>
<td>&lt; 0.0002</td>
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<td>&lt; 0.02</td>
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<tr>
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<td>CC / CC</td>
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<td>0.2</td>
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<td>AM</td>
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<td>(0.51)</td>
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<td>&lt; 0.01</td>
<td>&lt; 0.02</td>
<td>(8.0)</td>
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<td>&lt; 0.2</td>
<td>&lt; 0.02</td>
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<td>1.2</td>
<td>&lt; 0.2</td>
<td>&lt; 0.02</td>
<td>0.63</td>
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<td>&lt; 0.02</td>
<td>&lt; 0.02</td>
<td>&lt; 0.0002</td>
<td>&lt; 0.2</td>
<td>&lt; 0.02</td>
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<td>AM</td>
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<td>&lt; 0.01</td>
<td>&lt; 0.02</td>
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<td>&lt; 0.02</td>
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<td>JL / MK</td>
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<td>PM</td>
<td>&lt; 0.2</td>
<td>1.1</td>
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</table>

**NUMBER OF SAMPLES**
- Average: 14
- Average: 14
- Average: 14
- Average: 14
- Average: 14
- Average: 14
- Average: 14

**CI = AVERAGE + T.*STD ERROR**
- CI = 0.200 + 0.048
- CI = 0.398
- CI = 0.335
- CI = 4.753
- CI = 0.0002

**Reporting Limit:**
- 500
- 1000
- 1.00

**Ash Moisture Test Result**
- Date: 2/18/2015
- Comments: Test completed as part of the Quarterly Residue Testing, no free liquids present.

**RECEIVED - DEQ**
- APR 10 2015
- Tidewater Regional Office
April 29, 2016

Jeff Delbier
Waste Compliance Technical Coordinator
Virginia Department of Environmental Quality
5636 Southern Blvd.
Virginia Beach, VA 23462

Re: Wheelabrator Portsmouth WTE Ash Test Results

Dear Mr. Delbier,

In accordance with 9VAC20-81.340.E.7 please find enclosed a copy of the chemical analyses of residue completed in March 2016.

If you have any questions about this submittal, you can reach me at 393-3105.

Sincerely,

Jeff Landrum
Environmental Manager

Enclosure
DOCUMENT CERTIFICATION

Facility Name: Wheelabrator Portsmouth Inc.
Registration No. 61018
Facility Location: Portsmouth, VA
Type of Submittal Attached: 2016 Residue Chemical Analysis Results

Certification: I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering and evaluating the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

Name of Responsible Official (Print): Rob Johnson
Title: Plant Manager
Signature: [Signature]  Date: 04/21/2016
## Wheelabrator Portsmouth Inc
### Inorganic Analysis Report

Residue Chemical Analysis Report

Report Date: 4/12/2016

<table>
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<th>SAMPLE ID</th>
<th>SAMPLED BY</th>
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<th>AM/PM</th>
<th>As (mg/L)</th>
<th>Ba (mg/L)</th>
<th>Cd (mg/L)</th>
<th>Cr (mg/L)</th>
<th>Pb (mg/L)</th>
<th>Hg (mg/L)</th>
<th>Se (mg/L)</th>
<th>Ag (mg/L)</th>
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<td>6014</td>
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<td>3/13/2016</td>
<td>AM</td>
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<td>&lt; 1.5</td>
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<td>6025</td>
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</table>

### Number of Samples
- 14

### Average
- As: 0.500
- Ba: 1.193
- Cd: 0.561
- Cr: 0.052
- Pb: 0.517
- Hg: 0.0002
- Se: 0.500

### Standard Deviation
- 0.00

### Std Error
- 0.00

### T Test
- 0.00

### CI = Average + T.20(Std Error)
- 0.500

### Reporting Limit
- 5.00

---

### Ash Moisture Test

<table>
<thead>
<tr>
<th>Date</th>
<th>Result</th>
<th>Comments</th>
</tr>
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<tbody>
<tr>
<td>3/16/2016</td>
<td>Pass</td>
<td>Test completed as part of the Quarterly Residue Testing, no free liquids present</td>
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</tbody>
</table>

4/12/2016
April 8, 2013

Jeff Deibler
Waste Compliance Technical Coordinator
Virginia Department of Environmental Quality
5536 Southern Blvd.
Virginia Beach, VA 23462

Re: Wheelabrator Portsmouth WTE Ash Test Results

Dear Mr. Deibler,

In accordance with 9VAC20-61-340.E.7 please find enclosed a copy of the chemical analyses of residue completed in March 2013.

If you have any questions about this submittal, you can reach me at 393-3105.

Sincerely,

[Signature]

Jeff Landrum
Environmental Manager

Enclosure
DOCUMENT CERTIFICATION

Facility Name: Wheelabrator Portsmouth Waste to Energy Facility

Facility Location: Portsmouth, VA

Type of Submittal Attached: March 2013 Residue Chemical Analysis Results

Certification: I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering and evaluating the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

Name of Responsible Official (Print): Paul Greene

Title: Plant Manager

Signature: [Signature]

Date: 04/08/2013
## Inorganic Analysis Report

### Sampling Event (in mg/L)

<table>
<thead>
<tr>
<th>SAMPLE ID</th>
<th>SAMPLED BY</th>
<th>DATE</th>
<th>ASP</th>
<th>HA</th>
<th>Cd</th>
<th>Cr</th>
<th>Pb</th>
<th>Hg</th>
<th>Se</th>
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<td>3/4/2013 AM</td>
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<td>&lt; 1</td>
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<td>2.3</td>
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### Numerical Data

- **Number of Samples**: 14
- **Average**: 0.500
- **Std Deviation**: 0.010
- **Std Error**: 0.001
- **T-20**: 1.35
- **CI = Average + T-20(Std Error)**: 0.500

### Reporting Limit

- **5.00 mg/L**

---

### Ash Moisture Test

- **Date**: 3/7/2013
- **Result**: Pass
- **Comments**: Test completed as part of the Quarterly Residue Testing, no free liquids present.
VITA

Anil Mehrotra, M.S., P.E., BCEE
E-mail: amehro002@odu.edu
mehrotraa@tncc.edu

Education:
D. Eng. candidate, CEE, Old Dominion Univ., expected completion summer 2017
M.S. Environmental Engineering, Old Dominion Univ., Norfolk
2 Graduate Courses (8 credits), Environmental Management, Harvard University
B.S. Mechanical Engineering, IIT, Roorkee (previously Univ. of Roorkee), India
B.S. (Math, Physics, Chemistry), Agra College, India

Licenses:
P.E. License 0402 043969
Board Certified Environmental Engineer (American Academy of Env. Engineers)
Advanced Engineering in Energy Systems Certificate, ODU, fall 2014
CPE, Certified Plant Engineer Certificate, from Association of Facilities Engineers
ASME QRO (Qualified Resource Operator) License, P-1745
Virginia Waste Facility Operators Class I-Class IV License, 460-5002344
Virginia Master Naturalist Certificate, VCE, Virginia Tech, Cohort 4, 2012

Professional Experience: Over 48 yrs. in conventional and alternative energies, combustion
   technologies, plant operations and maintenance, environmental
ingineering.

Current Employment:
Plant Engineer, Hampton/NASA Steam Plant 2001 to date
Industrial plant systems engineering design, upgrades and management
Environmental audits for effectively maintaining facility’s following permits:
1. Title V Air permit,
2. Municipal Solid Waste permit for waste processing, ash treatment & disposal,
3. Wastewater discharge permit

Knowledgeable in the Clean Air Act, National Pollutant Discharge Elimination
System (NPDES) regulations, Resource Conservation and Recovery Act (RCRA),
Superfund and Amendments and Reauthorization Act (SARA Tier II), Safe
Drinking Water Act (SDWA), Spill Prevention, Control, and Countermeasure
(SPCC)

Employee training in standards of plant engineering and operations
Manager, Safety & Health for compliance with OSHA regulations and safety
audits

Previous Experience:
High capacity energy plants (India): Toshiba, GE, Mitsubishi, CE model
   turbines/boilers
High pressure boilers & turbines designs, operation, maintenance 1968 – 1973
District energy supply, power distribution network designing 1974 - 1979
Heavy machinery dynamics, vibration analysis, performance testing 1980 - 2000
Designing and engineering, project scheduling, start-up, environmental
management, budgeting, issuing RFP, vendor selection, awarding and managing
contracts, operation and Maintenance of power generation equipment, leadership and organizational management.
Developing and teaching industrial operating standards & technical training modules

**Professional Memberships:**

- Member, State Advisory Board, VA Air Pollution Control Board  Since 2010
- Water Environment Federation, International Coordination Committee  Since 2012
- American Society of Safety Engineers  Since 2007
- National Safety Council  Since 2007
- ASME International  Since 2001

**Research Papers and Professional Skills Development:**

- Doctoral research on immobilization and treatment of municipal solid waste incineration residues, Old Dominion University, June, 2017
- Sustainable Use of Fibrous Natural Resources- Treatment Methods for Increased Utilization of Bamboo, August, 2015
- Heat and Energy Recovery from Biosolids, M.S. Thesis, Old Dominion University, May, 2015
- Adjunct Faculty, Science Engineering and Technology, Thomas Nelson Community College, Hampton, Virginia since fall 2015
- Thermal Treatment of Municipal Solid Waste to Produce Biofuels, Independent Study, Old Dominion University, June, 2013
- City of Hampton facility representative for ASME and EPA Air Pollution Control Technologies and Regulations, Emission Guidelines and GHG Reporting Tools
- District Energy Systems – Improving Efficiency for Producing Electricity, Heating and Cooling Building Complexes, Participant Member, Va. State Advisory Board, Nov. ‘12
- OSHA General Industry 10-hr and 32-hr Safety and Health training, attended Department of Labor and Industries, and Federal Emergency Management courses.
- Instructor since November 2011 Waste Management Facility Operator Class I Course Training to City of Hampton employees, and other waste processors in Virginia.
- Fine Particle Air Pollution –Respiratory health issues concerning at risk population in Hampton Roads, Southeastern Virginia, Environmental Management, Harvard University Extension School, July 1, 2011.
Author, Waste Management Facility Operators Course Modules, Approved Training provider, Department of Professional and Occupational Regulations, Virginia, July 17, 2011
Climate Change Mitigation and Adaptation in the Commonwealth, Member State Advisory Board on Air Pollution, Va. State Air Pollution Control Board, Dec, 10.
Reducing Compliance Costs through Modernized Reporting, Principal Contributor, State Advisory Board Compliance Cost Group, December, 2010.
Green House Gas Option Rating Priority Summary, State Advisory Board on Air Pollution, Virginia State Air Pollution Control Board, December 2010.
Paper, Deformation and Integrity of Structural Components at Elevated Temperatures, Institute of Technology, Banaras Hindu Univ., Varanasi, UP, India, 1998
Paper, Condition Monitoring and Modifications to Control Stray Voltages in 500 MW Turbine-Generator Shafts, Anpara B Thermal Power Station, UPSEB (now U.P. Power Corporation Ltd.), India, Nov. 10, 1995