STANDARD OPERATING PROCEDURE FOR:

TOTAL SUSPENDED SOLIDS DETERMINATION USING VACUUM FILTRATION AND OVEN DRYING

TVA-KIF-SOP-49

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for
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1.0 PURPOSE

This standard operating procedure (SOP) provides the general technical requirements and operational guidelines for determining total suspended solids (TSS) of process water collected during dredging activities at Kingston Fossil Plant (KIF) Ash Recovery Project Site using vacuum filtration and oven drying.

This SOP is in support of the *Kingston Fly Ash Recovery Project Monitoring of Water Discharged During Dredged Fly Ash Dewatering Operations,* Work Plan Update (Jacobs, 2009) and is based on American Society for Testing of Materials (ASTM) D 5907-09 *Standard Test Method for Filterable and Nonfilterable Matter in Water.*

2.0 GENERAL CONSIDERATIONS

Potential hazards associated with the planned tasks are thoroughly evaluated prior to conducting field activities. The *Site-Wide Safety and Health Plan* (SWSHP) provides a description of potential hazards and associated safety and control measures.

A job safety analysis (JSA) was performed for this procedure and is included as Table 1 of this SOP. Per the JSA included, laboratory ovens have hot internal surfaces. Personnel wear appropriate gloves when removing pans and items from the drying ovens and avoid touching internal surfaces. For other operations, personnel wear powder-free nitrile gloves. Specifically, powder-free nitrile gloves are worn while handling the samples during moisture content analyses.

3.0 PROCEDURES

This section documents the equipment required, general operating procedures, and methods associated with determining the total suspended solids of water samples at KIF using vacuum filtration and an oven drying.

3.1 Equipment

The equipment required for performing these procedures includes balances and drying ovens and the following items.

- Buchner Funnel – A ceramic funnel with a flat or fitted base that provides uniform support and a uniform, filterable surface.
- Glass fiber filter – Whatman 934-AH glass microfiber filters of adequate size for use with the chosen funnel (such as a Buchner Funnel with a diameter of 110 mm requires 110 mm filter paper).
- Vacuum Filter Flask – Glass flask equipped with a side arm for vacuum pump connection and a rubber stopper to provide seal with funnel. The flask is used to collect the filtered process water that passes through the filter.

- Vacuum Pump – a suitable vacuum is required for the filtering of process water through the funnel and filter paper

Procedures for the use and calibration of the balances and ovens are defined below.

3.1.1 Balances

Both the precision and analytical balances are used in support of this procedure. The analytical balance is used to determine the weight of the glass microfiber filter paper used for this method prior to filtering and after the drying step (Section 3.6) has been completed. A balance that weighs to 0.001 g is required for specimens having a weight up to 5.00 g. The precision balance is used for the determination of the percent solids by weight of samples with an estimated TSS greater than 20,000 mg/L.

The balance calibration and daily verification is performed in accordance with *Daily Calibration Checks for Balances at the KIF Field Laboratory* SOP (TVA-KIF-SOP-48).

3.1.2 Drying Oven

A vented, thermostatically-controlled unit capable of maintaining uniform temperature throughout the drying chamber is used.

a. Set the drying temperature of the oven at 110 ± 5°C using a NIST traceable thermometer or thermocouple.

b. Monitor temperature of the drying oven with a NIST-certified or traceable thermometer.

c. Record temperature in the drying oven logbook each morning and evening that the oven is in use.

3.2 Sample Homogenization and Test Aliquot

The samples to be analyzed are process water from dredging activities during the removal of ash from the Emory River. The samples are collected from areas designated by the *Kingston Fly Ash Recovery Project Monitoring of Water Discharged During Dredged Fly Ash Dewatering Operations* Work Plan Update (Jacobs 2009). Prior to analyzing the samples for TSS, each one must be homogenized by stirring, shaking or mixing the sample container thoroughly prior to measuring the volume needed for filtration.

The volume of sample needed must yield between 2.5 and 500 mg dried residue. Based on estimated visual observations of approximated TSS values and/or in comparison to a reference TSS sample set (located in the field laboratory), the technician analyzing the samples uses judgment to adjust the sample volume required .

The following table provides the historical volumes used for TSS analysis at the different sample locations during typical dewatering conditions for the KIF Ash Recovery Project.

Sample Aliquot Size for TSS Analysis

Sample Location(s)	Approximate TSS (mg/L)	Volume Required for Analysis (mL)
Stilling Pond Ash Pond Filter Press Filtrate	0 – 100	1000
Lateral Expansion	100 – 1000	250 to 500
Rim Ditch Sluice Trench	1000 – 20,000[1]	100

[1] Samples with an estimated TSS >20,000 mg/L are also analyzed for percent solids by weight per Section 3.4 of this procedure.

3.3 Determination of Total Suspended Solids

The following steps provide the procedure for determining TSS of process water samples. There are separate worksheets for recording the TSS sample results collected in the morning and evening provided as Tables 2 and 3.

a. Wash and dry filters for this analysis prior to use by rinsing the filter with deionized (DI) water three times while vacuum is applied through the vacuum filtration assembly and place in a drying oven with a temperature of 110 ± 5°C for a minimum of one hour.

b. Record the weight of the pre-washed and prepped filter on the appropriate worksheet (Tables 2 or 3).

c. Record the sample collection time, pH, and temperature from the field logbook on the appropriate worksheet (Tables 2 or 3).

d. Assemble filtering apparatus by placing the filter paper in a cleaned Buchner funnel and begin suction.

e. Wet the filter with a small volume of reagent-grade water to seat it.

f. Shake, stir, or mix the contents in the sample container in order to obtain a more uniform (preferably homogeneous) aliquot for analysis.

g. Immediately after homogenizing the sample, collect the required aliquot (see the above table in Section 3.2 for the appropriate volume) in a clean graduated cylinder. Record the sample aliquot volume on the appropriate worksheet (Tables 2 or 3).

h. Slowly pour the volume onto the seated glass microfiber filter until the entire aliquot has been filtered. If residue lingers in the cylinder, rinse the cylinder with DI water and filter this rinse water.

i. After filtering the sample aliquot, wash the Buchner funnel clean of any residual sample with DI water, allowing complete drainage between washings, and continue suction until filtration is complete.

j. Carefully remove filter from filtration apparatus using forceps, tweezers, or tongs, and transfer to an aluminum or glass weighing dish as a support.

k. Dry filters for a minimum of 1 hour at 110 ± 5°C in drying oven.

l. After drying is complete, reweigh the filters and record the dry filter and residue weight on the appropriate worksheet (AM or PM) included as Tables 2 or 3.

m. Use the following calculation to determine TSS.

$$TSS\ \left(\frac{mg}{L}\right) = \frac{(Residue + filter)(mg) - Filter\ (mg)}{Sample\ filtered\ (mL)} x1000(\frac{mL}{L})$$

3.4 Determination of Percent Solids by Weight

Samples with an estimated TSS >20,000 mg/L are analyzed for percent solids by weight in addition to TSS. Historically, this test has only been applicable to the Rim Ditch, Sluice Trench, and Lateral Expansion samples.

a. Weigh a large aluminum pan and record the tare weight on the appropriate worksheet (Tables 2 or 3).

b. Pour 60 ml of sample into the aluminum pan and record the weight of the pan and sample on the worksheet.

c. Place pan and sample in the drying oven at 110 ± 5°C until the sample is completely dry (at least two hours).

d. Remove pan from the oven and weigh the pan and dried sample. Record the weight of the dried sample and pan on the worksheet.

e. Use the following calculation to determine percent solid by weight.

$$Percent\ Solids = \frac{\text{Dry mass of sample (Container + dry sample mass – container mass)}}{\text{Wet Mass of sample (Container + wet sample mass – container mass)}}\ x\ 100$$

3.5 Daily Quality Control Sample – Blank

A sample blank accompanies the TSS analysis at a frequency of one each day. The blank filter will be run according to Section 3.3 above where 250 ml of DI water replaces the sample aliquot as required in Step *g*.

3.6 Laboratory Logbooks and Documentation

Field logbooks are maintained by the sampling and laboratory personnel and used to record field sampling activities. The field logbooks will contain information regarding the sampling events per the requirements discussed in the *Field Documentation* SOP (TVA-KIF-SOP-06). Daily calibration verification of the balances and the daily oven temperature monitoring are recorded in logbooks dedicated to each instrument. Laboratory TSS worksheets are used to document the sample data and calculations: *Morning/AM Total Suspended Solids & %Moisture* (Table 2) and *Evening/PM Total Suspended Solids & %Moisture* (Table 3).

The Field Laboratory Supervisor and/or designee reviews the field logbook, laboratory logbooks, and data worksheet entries on a weekly basis at a minimum (daily review is preferred) for completeness and accuracy and indicate this review by initialing the entries.

3.7 Decontamination and Waste Management

The water that has been processed and analyzed is managed in accordance with the *Management of Investigation-Derived Waste* SOP (TVA-KIF-SOP-12). The filters and disposable PPE are managed as ash-contaminated waste, stored in plastic trash bags, and disposed with the other TVA-KIF site ash-contaminated waste in accordance with the TVA-KIF-SOP-12.

4.0 REFERENCES

- American Society for Testing and Materials (ASTM). ASTM D5907-09, *Standard Test Method for Filterable and Nonfilterable Matter in Water*, www.astm.org/Standards/D5907.htm.
- Jacobs. *Kingston Fly Ash Recovery Project Monitoring of Water Discharged During Dredged Fly Ash Dewatering Operations,* Work Plan Update, 2009.
- Tennessee Valley Authority (TVA). *Daily Calibration Checks for Balances at the KIF Field Laboratory* (TVA-KIF-SOP-48), 2010.
- TVA. *Field Documentation* SOP (TVA-KIF-SOP-06), 2009.
- TVA. *Determining Moisture Content of Ash Samples Using Halogen Moisture Analyzer* SOP (TVA-KIF-SOP-44), 2010.

- TVA. *Determining Moisture Content of Ash Samples Using Oven Drying Method* SOP (TVA-KIF-SOP-49), 2010
- TVA. *Management of Investigation-Derived Waste* SOP (TVA-KIF-SOP-12), 2010.
- TVA. *Site-Wide Safety and Health Plan for the TVA Kingston Fossil Plant Ash Release Response* (SWSHP), Jacobs, June 2009.

Table 1. Job Safety Analysis

Activity/Task: Laboratory Testing **Work Area:** TVA KIF Field Laboratory

Date _____

Steps of Activity/Task	Potential Hazard	Safe Plan	Resources
General lab housekeeping	Crowded space with many supplies and instruments in small area.	Keep area neat and clean with supplies stored on shelves above working area. Keep walkways and floor areas free of equipment and supplies.	Shelves and other side of trailer available for storage.
	Lifting buckets of soils or coolers	Use proper lifting practices and a buddy to lift heavy coolers	
	Supplies stored above workspace.	Use step stool to access supplies stored above work space. Do not stand on chairs or tables to reach items above work space.	Step stool
Performing lab tests (moisture content, TSS, etc.)	Exposure to ash and aqueous samples collected from the ballfield and ash recovery system.	Wear safety glasses at all times in the laboratory. Wear gloves at all times when handling samples or equipment that comes in contact with samples.	Nitrile gloves, safety glasses, and eyewash available in lab.
	Glassware breakage and subsequent cuts and abrasions	Handle glassware carefully. Always discard or remove glassware from service if chipped, cracked or otherwise compromised.	
Using drying oven and moisture analyzers for moisture and TSS analyses.	Very hot inside oven and inside the moisture analyzers (110 degrees C). Pinch points around door of oven and lid for moisture analyzers.	Use gloves suitable for handling hot items when removing or adding samples to the oven and moisture analyzers. Keep hands free of pinch points around oven door and lids of moisture analyzers.	Use Teflon coated gloves or liners available in lab.

Steps of Activity/Task	Potential Hazard	Safe Plan	Resources
pH testing	Buffer solutions used for pH meter calibration.	Always wear proper PPE when using chemicals. Keep solutions stored in labeled bottles with lids secured when not in use.	MSDS
Use of TVA vehicle for sample collection and transport.	Low fluid levels or other malfunctioning equipment.	Complete daily walk around inspection of vehicle.	Document in logbook.

Signatures of Persons Involved in Review of JSA

_____

_____

_____

_____

_____

_____

(Supervisor signature/date)

Work shall stop when conditions change, the job changes, or a deficiency is discovered, and the current JSA will be modified or new JSA created.

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TABLE 2. MORNING/AM TOTAL SUSPENDED SOLIDS & % SOLIDS

SAMPLE DATE			
TEST DATE			
TESTED BY			
PAGE	1	OF	1
		PROJECT No.	10504

[ ] **Fill in grayed out boxes only**

Staff gauge at stilling pond:	
Staff gauge at ash pond:	

NOTES:

Ash Pond Polymer Tanks:	full
Sluice Trench Polymer Tanks:	full
Rim Ditch Polymer Tanks:	full

SAMPLING CONDITIONS:

Test Data

Sample ID	SP-(MMDDYY)-A	AP-(MMDDYY)-A	STE-(MMDDYY)-A	RDE-(MMDDYY)-A	FPE-(MMDDYY)-A	--
Sample Location	Stilling Pond AM	Ash Pond AM	Sluice Trench Effluent AM	Rim Ditch Effluent AM	Filter Press Filtrate AM	BLANK
Collection Time of Sample						--
Temperature (°F)						--
pH						--
Sample Volume [ml]			**	**		
Filter Paper mass [grams]						
Dry Filter and Solids [grams]						
Dry Solids mass [grams]	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Solids by TSS [mg/l]						

****Note: If TSS >20, 000 mg/L predicited, oven method will be used (See % Solilds (by weight) Table below). If questionable (>20,000) BOTH the filter method and the oven method will be used.**

% Solids (by weight)

Sample ID	Container Mass [grams]	Volume [ml]	Container + Wet Sample Mass [grams]	Wet Sample Mass [grams]	Container + Dry Sample Mass [grams]	Dry Solids Mass [grams]	**% Solids (by weight) [%]**	**Approximate TSS mg/L**
STE-(MMDDYY)-A				0.00		0.00		
RDE-(MMDDYY)-A				0.00		0.00		

Filter Cake Sample from Filter Press

Sample ID	Collection Time	Al Pan Wt [grams]	Al Pan + Sample Wet Weight [grams]	Wet Sample Mass [grams]	Al Pan with Dried Sample [grams]	Solids Mass [grams]	**% Solids (by weight) [%]**	**Moisture (Moetler Toledo HB 43S) [%]**
Filter Cake				0.00		0.00		

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TABLE 3. EVENING/PM TOTAL SUSPENDED SOLIDS & % SOLIDS

SAMPLE DATE			
TEST DATE			
TESTED BY			
PAGE	1	OF	1

Fill in grayed out boxes only

TVA/KIF Ash Recovery	PROJECT No. 10104
NOTES: Ash Pond Polymer Tanks:	full
Sluice Trench Polymer Tanks:	full
Other:	
SAMPLING CONDITIONS	

Staff gauge at stilling pond:	
Staff gauge at ash pond:	

Note: If TSS >20, 000 mg/L predicited, oven method will be used. If questionalbe (>20,000) filter method and oven method will be used.

Test Data

Sample ID	SP-(MMDDYY)-P	AP-(MMDDYY)-P	STE-(MMDDYY)-P	RD-(MMDYY)-P	LE-(MMDYY)-P
Sample Location	Stilling Pond PM	Ash Pond PM	Sluice Trench Effluent PM	Rim Ditch PM	Lateral Expansion PM
Collection Time of Sample					
Temperature (^{o}F)					
pH					
Sample Volume [ml]					
Filter Paper mass [grams]					
Dry Filter and Solids [grams]					
Dry Solids mass [grams]	0.0000	0.0000	0.0000	0.0000	0.0000
Solids by TSS [mg/l]					

Sample ID	Container Mass [grams]	Volume [ml]	Container + Wet Sample Mass [grams]	Wet Sample Mass [grams]	Container + Dry Sample Mass [grams]	Dry Solids Mass [grams]	**% Solids (by weight) [%]**	**Approximate TSS mg/L**
STE-(MMDDYY)-P				0.00		0.00	**NA**	**NA**
RD-(MMDYY)-P				0.00		0.00	**NA**	**NA**

END OF PROCEDURE