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VERSO CORPORATION
Quinnesec Mill

SITE-SPECIFIC FUEL SAMPLING & ANALYSIS PLAN

FOR 40 C.F.R. PART 63, SUBPART DDDDD
(NESHAP for Industrial, Commercial, and Institutional Boilers and Process Heaters)

September 2017

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1.0 INTRODUCTION

Verso Corporation's Quinnesec Mill is a coated and publication paper manufacturing facility located in Quinnesec, MI. The facility is subject to *40 C.F.R. Part 63 National Emission Standards for Hazardous Air Pollutants for Industrial, Commercial, and Institutional Boilers and Process Heaters* (hereafter referred to as Boiler MACT). The Boiler MACT allows the use of fuel analysis or performance (stack) testing to demonstrate compliance with the mercury (Hg), hydrogen chloride (HCl), carbon monoxide (CO), and total selected metals (TSM) emission limits. Regardless of the compliance demonstration method selected, each facility subject to the Boiler MACT must develop and then submit (if requested, or required if using alternative methods) a site-specific fuel analysis plan in accordance with 40 C.F.R § 63.7521(b).

Fuel Sampling Requirements

The Quinnesec Mill will demonstrate compliance with the applicable Hg and HCl emission limits using performance testing. This requires a fuel analysis to be conducted during the initial performance (stack) test or subsequent performance test if a new fuel type is burned with a higher pollutant loading than that which was established during the initial performance test.

In the event that the Quinnesec Mill chooses to demonstrate compliance with the applicable Hg and HCl emission limits via monthly fuel analysis, the procedures outlined in this plan will be used.

For reference, Table 1-1 provides the required elements for the site-specific fuel analysis plan.

Table 1-1 Cross Reference of Site-Specific Fuel Analysis Plan Requirements

Regulatory Citation & Description		**Plan Section Number**
§63.7521(b) (1) You must submit (if requested) the fuel analysis plan no later than 60 days before the date that you intend to demonstrate compliance if alternative methods are used.		2.0
§63.7521(b) (2) You must include the information contained in paragraphs (b) (2) (i) through (vi) of this section in your fuel analysis plan.		
(2)(i)	The identification of all fuel types anticipated to be burned in each boiler or process heater.	3.0
(2)(ii)	For each fuel type, the notification of whether you or a fuel supplier will be conducting the fuel analysis.	5.1
(2)(iii)	For each fuel type, a detailed description of the sample location and specific procedures to be used for collecting and preparing the composite samples if your procedures are different from paragraph (c) or (d) of this section. Samples should be collected at a location that most accurately represents the fuel type, where possible, at a point prior to mixing with other dissimilar fuel types.	4.1
(2)(iv)	For each fuel type, the analytical methods, with the expected minimum detection levels, to be used for the measurement of hydrogen chloride, or mercury.	5.2
(2)(v)	If you request to use an alternative analytical method other than those required by Table 6 to this subpart, you must also include a detailed description of the methods and procedures that will be used.	5.2
(2)(vi)	If you will be using fuel analysis from a fuel supplier in lieu of site-specific sampling and analysis, the fuel supplier must use the analytical methods required by Table 6 to this subpart.	N/A

2.0 PLAN SUBMITTAL REQUIREMENT

40 C.F.R.§ 63.7521(b)(1) requires that a facility submit the fuel analysis plan no later than 60 days before the date you intend to demonstrate compliance if alternate test methods other than those in Table 6 of the Boiler MACT rule are used.

Fuel Sampling Required during Performance Testing

For initial performance testing or performance testing to demonstrate compliance burning new fuel(s) the Quinnesec Mill will conduct fuel sampling on the date of the performance test when demonstrating compliance with the emission standards for mercury (Hg) and hydrogen chloride (HCl) in Table 2 of the MACT rule. The Quinnesec Mill will be sending the samples to a laboratory that will be using equivalent sample preparation and test methods other than those explicitly listed in Table 6 of the rule.

Each composite fuel sample collected during the performance test runs will be made up of three individual samples . Samples collected from a pile will consist of three composite samples collected according to 63.7521(c)(2)(i) through (iii).

Fuel Sampling Required for Monthly Fuel Analysis

In the event that the Quinnesec Mill chooses to conduct monthly fuel sampling for compliance, fuel sampling would start after January 2016. By submitting the fuel analysis plan at the time of initial performance testing, the 60-day notification requirement for alternative/equivalent methods has been met.

3.0 BOILER AND FUEL INFORMATION

The Quinnesec Mill operates a multi-fuel fired boiler (Waste Fuel Boiler or WFB) which is subject to the Boiler MACT standard. The WFB produces steam for energy generation and provides heat for the pulp and paper-making process. A natural gas fired package boiler is also an affected unit under the rule with no fuel sampling requirements. An initial notification for these boilers was submitted to EPA and MDEQ (Michigan Department of Environmental Quality) on May 14, 2013. A description of the WFB and the fuel types burned are provided in Table 3-1.

Table 3-1 Quinnesec Mill Boiler Description and Fuel Types

Boiler ID	Description	Maximum Rated Capacity	Fuels	Control Device (Installation Year)
WFB (EU1121)	Installed: 1985 Manufacturer: B&W Boiler Category: Stoker/Sloped grate designed to burn wet biomass	660 MMBtu/hr 363 MMBtu/hr fossil fuel limit	Biomass[1] Coal Natural Gas[2]	Electrostatic Precipitator Installed: 1985

[1] Biomass includes bark, sawdust, clean wood debris wood residue; wood products (*e.g.,* trees, tree stumps, tree limbs, lumber, sander dust, chips, scraps, slabs, millings, and shavings.

[2] Per § 63.7510 (a)(2)(ii), when natural gas or other gas 1 fuels are co-fired with other fuels, you are not required to conduct a fuel analysis of natural gas or other gas 1 fuels according to § 63.7521 and Table 6 to Subpart DDDDD.

4.0 FUEL SAMPLING

The specific sampling and preparation procedures listed in 40 CFR §63.7521(c) and (d) are included in Appendix A and will serve as the protocol for all solid fuel samples. Natural gas (gas 1 fuel) is exempt from monitoring requirements as specified in §63.7510(a)(2)(ii).

4.1 SAMPLE LOCATIONS AND PROCEDURES

Biomass

Quinnesec Mill personnel will conduct on-site fuel sample collection from the facility's boiler bark/wood feed conveyor according to the procedures specified in §63.7521(c)(2) For analysis as part of a performance test, a composite sample will be collected for each test run. If collected for monthly fuel analysis, a minimum of one composite sample will be collected. The analyzing laboratory will prepare the composite samples as specified in §63.7521(d).

Coal

Quinnesec Mill personnel will conduct on-site fuel sample collection from the facility coal pile according to the procedures specified in §63.7521(c)(2). For analysis as part of a performance test, sampling from the coal pile will be conducted in the hours prior to or during the performance test. Because the coal supply pile does not change throughout the performance testing, sampling will not be conducted during each run. If collected for monthly fuel analysis, a minimum of one composite sample will be collected. The analyzing laboratory will prepare the composite samples as specified in §63.7521(d).

4.2 GENERAL REQUIREMENTS FOR MONTHLY FUEL ANALYSIS

If monthly fuel analysis is used as the compliance method, a minimum of one composite sample will be collected during the month for each fuel type combusted. The monthly fuel sampling and preparation will follow the procedures outlined above. Fuel analysis may be conducted any time within the calendar month as long as the analysis is separated from the previous analysis by at least 14 calendar days. If a new type of fuel is burned, fuel analysis will be conducted before burning the new type of fuel. If each of 12 consecutive monthly fuel analyses demonstrates 75 percent or less of the compliance level, the fuel analysis frequency may be decreased to quarterly for that fuel. If any quarterly sample exceeds 75 percent of the compliance level a new type of fuel is burned, monthly monitoring will be resumed for that fuel, until 12 months of fuel analyses are again less than 75 percent of the compliance level. If sampling is conducted on one day per month, samples will be collected no less than 14 days apart. If multiple samples are taken per month, the 14-day restriction does not apply.

5.0 FUEL ANALYSIS

5.1 ANALYTICAL LABORATORY

All samples collected by the Quinnesec Mill's personnel will be sent to ALS Life Sciences Division, Environmental 3860 South Palo Verde Road, Suite 302 Tucson, AZ 85714 USA, www.alsglobal.com for sample preparation and analysis. ALS Tucson has received accreditation from the American Association for Laboratory Accreditation for fuel sampling which meets the sampling requirements in Boiler MACT. Appendix B provides a copy of their accreditation (Copies of new certification will be periodically required.)

Samples may alternatively be sent to the following lab: ALS Environmental 9143 Philips Highway, Jacksonville, FL 32256, 904-739-2277 www.alsglobal.com in Jacksonville, FL. ALS Jacksonville has received NELAP accreditation through the State of Florida Department of Health, Bureau of Laboratories for several analytical techniques including: extractable organics, general chemistry, metals and volatile organics—all from under the solid and chemical materials category.

Analysis of new fuels or non-performance test fuels used to demonstrate ongoing compliance with established pollutant loading values will be conducted by the Quinnesec Mill or the fuel supplier. Samples collected by Quinnesec Mill's personnel will be collected utilizing the appropriate method specified at §63.7521(c). All samples will be will be analyzed by a contract lab or the supplier using the methods or equivalent methods as specified in Table 6 of 40 CFR 63 Subpart DDDDD.

5.2 ANALYTICAL PROCEDURES

Table 5-1 below contains the list of relevant sample collection, preparation, and test methods from Table 6 to Subpart DDDDD of Part 63—Fuel Analysis Requirements (see the column labeled Table 6 method); these methods have been reviewed and approved by the Environmental Protection Agency (EPA). Table 5-1 also shows the Equivalent Methods that will be used by the mill (see the last column in the table below). Additional descriptions of the equivalent/alternative methods are included in the footnotes below.

Table 5-1: Fuel Analysis Test Methods

To conduct a fuel analysis for the following pollutant . . .	You must . . .	Table 6 Method[3]	Equivalent /Alternative Methods Used by the Mill
1. Mercury	a. Collect fuel samples	Procedure in § 63.7521(c) or, or ASTM D4057 (for liquid), or equivalent.	The bark samples are collected and temporary transferred to a clean plastic bucket. Once three samples are collected, they are then mixed and quartered in accordance with § 63.7521(d). For liquid sampling, see the prior section 4.2 of this plan.
	b. Composite fuel samples	Procedure in § 63.7521(d) or equivalent.	-
Mercury	c. Prepare composited fuel samples	…or equivalent[4]	Equivalent method: ASTM E1757[5] for biomass and sludge Liquid fuel samples do not need preparation, other than mixing prior to analysis.

[3] Additional methods are listed in Table 6 of the rule.

[4] In Table 6, the only listed method that details how to prepare a fuel sample for analysis, including air drying and grinding, is ASTM D2013/2013M. Methods EPA SW-846-3050B, EPA 3050, and ASTM D5198 are acid digestion methods and do not cover the homogenization of raw bulk samples for analysis. Also, ALS believes these digestion methods are not appropriate for Mercury. Method EPA 821-R-01 does have some brief references for prepping biota and sludge samples for Mercury analysis, but not for solid matrix samples like biomass samples. (ALS)

[5] For ASTM E1757 "*Standard Practice for Preparation of Biomass for Compositional Analysis*", the sample preparation procedure is written to insure that samples are homogenized in a manner to maintain sample integrity by avoiding contamination of the sample, minimizing moisture loss and exposure to excessive heat during the grinding process while insuring that a representative sub sample is achieved that is suitable for analysis. The prep method involves air drying the raw sample at 40°C to minimize moisture loss during the preparation process. Air dried samples are the ground to < 1 mm using non-contaminating equipment were ever possible to homogenize the sample for analysis. The Air Dry Loss moisture (ADL) is determined from the weight lost during the air drying process and is used to calculate the Total Moisture. (ALS)

To conduct a fuel analysis for the following pollutant . . .	You must . . .	Table 6 Method[3]	Equivalent /Alternative Methods Used by the Mill
	d. Determine heat content of the fuel type	ASTM D5865[6] (coal), ASTM E711 (for biomass), or ASTM D240[7] or equivalent.	Equivalent Method: ASTM D5865 for biomass and sludge Equivalent Method: ASTM D4809[8] for liquids: *Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter*
Mercury	e. Determine moisture content of the fuel type	ASTM E871[9] , or ASTM D95 (for liquid fuels), or ASTM D4006 (for liquid fuels), or equivalent.	Equivalent method: ASTM D7582[10] for biomass and sludge. Moisture is not required for fuel oil to determine the heat content of the fuel. If required, method ASTM E1064 or D6869 [11] will be used.

[6] Methods D5865, E711 and D240 all use the same analytical technique and instrumentation – a LECO AC600 bomb calorimeter. The calibration of the bomb calorimeter is performed in the same manner using benzoic acid pellets in all methods. NIST traceable Standard Reference Material that are similar in matrix to the samples being analyzed and are used to verify the calibration of the calorimeter. The methods are equivalent except for matrix. (ALS)

[7] ASTM D240 "*Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter"*

[8] Method D4809 utilizes the same instrumentation as D240 and actually has better precision than D240.
[9] ASTM E871 *"Standard Test Method for Moisture Analysis of Particulate Wood Fuels"*

[10] ASTM D7582 **"***Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis*" is an automated method for the determination of Moisture, Volatile Matter, Fixed Carbon, and/or Ash. It is equivalent to ASTM methods D3173 and E871 for determining Moisture in that the samples are dried around 105C. The TGA system is automated so that the weights are measured repeated throughout the analytical run. The Moisture analysis is complete when the sample weights become constant and no additional moisture loss is measured. The equivalency of D7582 against D3173 and E871 is demonstrated by analyzing NIST traceable Standard Reference Materials as laboratory control samples with each run of 20 or fewer samples. ALS Tucson also participates in a quarterly in a round-robin program by analyzing and reporting blind proficiency standards. This information is available upon request from the laboratory (ALS).

[11] ASTM E1064 *"Standard Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration"*. ASTM D95 and D4006 methods use distillation. ASTM D6869 "*Standard Test Method for Coulometric and Volumetric Determination of Moisture in Plastics Using the Karl Fischer Reaction* (the Reaction of Iodine with Water)". Both methods use the Karl Fischer methodology.

To conduct a fuel analysis for the following pollutant . . .	You must . . .	Table 6 Method[3]	Equivalent /Alternative Methods Used by the Mill
	f. Measure mercury concentration in fuel sample	EPA SW-846-7471B (for solid samples), or EPA SW-846-7470A (for liquid samples), or equivalent.	Equivalent method: Mercury by D6722[12] for biomass, sludge, and liquid fuels
Mercury	g. Convert concentration into units of pounds of mercury per MMBtu of heat content	Equation 8 in § 63.7530.	-
2. HCl	a. Collect fuel samples	Procedure in § 63.7521(c) or, or ASTM D4057 (for liquid), or equivalent.	The bark samples are collected and temporary transferred to a clean plastic bucket. Once three samples are collected, they are then mixed and quartered in accordance with § 63.7521(d). For liquid sampling, see the prior section 4.2 of this plan.
	b. Composite fuel samples	Procedure in § 63.7521(d) or equivalent.	-
	c. Prepare composited fuel samples	…or equivalent	Equivalent methods will be used. See above. The same methods as for mercury will be used.
	d. Determine heat content of the fuel type	ASTM D5865 (coal), ASTM E711 (for biomass), or ASTM D240[a] or equivalent.	Equivalent methods will be used. See above. The same methods as for mercury will be used.
	e. Determine	ASTM E871, or ASTM D95	Equivalent methods will be used. See

[12] The Total Mercury method is performed following ASTM D6722 on a NIPPON MA3000 using high temperature combustion at 950°C with gold amalgamation to trap the Mercury followed by AAS to analyze the Mercury vapor that is released from the gold trap after it is rapidly heated. Method D6722 references the sample preparation method D2013 for coal, and D2013 references D3302 which involves Air Drying at 40°C prior to grinding samples for analysis. This method can be used for liquid and solid matrices. The method modification only pertains to the sample matrices that are not coal. Instrument calibration and the analytical processes are the same for all sample matrices. This technique is actually superior to EPA 7471B for solid matrices in that the analysis is direct and does not require the acid digestion step in 7471B. There are little to no matrix effects for D6722, it has better recovery of Mercury, and a lower reporting limit (down to 1 ppb for D6722) since there is no digestion step. The digestion step of 7471B can cause a low bias for samples with a complex matrix such as wood, refuse, tire derived fuel, and oil due to incomplete digestion of the sample (per ALS).
The performance of D6722 for each analytical run is measured by analyzing NIST traceable Standard Reference Materials with certified Mercury values that are similar in matrix as the samples. (ALS)

To conduct a fuel analysis for the following pollutant . . .	You must . . .	Table 6 Method[3]	Equivalent /Alternative Methods Used by the Mill
	moisture content of the fuel type	(for liquid fuels), or ASTM D4006 (for liquid fuels), or equivalent.	above. The same methods as for mercury will be used.
	f. Measure chlorine concentration in fuel sample	EPA SW-846-5050 or ASTM E776 (for solid fuel), or EPA SW-846-9056[13] or SW-846-9076 (for solids or liquids) or equivalent.	-
HCl	g. Convert concentrations into units of pounds of HCl per MMBtu of heat content	Equation 7 in § 63.7530.	-

Table 5-2 contains the test methods and expected detection limits for the fuels fired at the Androscoggin Mill.

Table 5-2 Fuel Analysis Detection Limits

Parameter	Test Method	Detection Limit
Hydrogen Chloride (Chlorine)	EPA SW-846-9056	100 ppm (solids & oil)
Mercury (Solids & Liquids)	ASTM D6722 mod	1 ppb (solids & oil)
Heat Content	ASTM D4809 (liquids) or ASTM D5865 (solids)	N/A
Moisture Content	ASTM D7582 Proximate by Automated TGA System	N/A

[13] Table 6 methods, 5050/9056, will be used for biomass and coal.

5.3 EQUIVALENCY

In accordance with § 63.7575, *Equivalent* means the following only as this term is used in Table 6 of the rule:

1. An equivalent sample collection procedure means a published voluntary consensus standard or practice (VCS) or EPA method that includes collection of a minimum of three composite fuel samples, with each composite consisting of a minimum of three increments collected at approximately equal intervals over the test period.
2. An equivalent sample compositing procedure means a published VCS or EPA method to systematically mix and obtain a representative subsample (part) of the composite sample.
3. An equivalent sample preparation procedure means a published VCS or EPA method that: Clearly states that the standard, practice or method is appropriate for the pollutant and the fuel matrix; or is cited as an appropriate sample preparation standard, practice or method for the pollutant in the chosen VCS or EPA determinative or analytical method.
4. An equivalent procedure for determining heat content means a published VCS or EPA method to obtain gross calorific (or higher heating) value.
5. An equivalent procedure for determining fuel moisture content means a published VCS or EPA method to obtain moisture content. If the sample analysis plan calls for determining metals (especially the mercury, selenium, or arsenic) using an aliquot of the dried sample, then the drying temperature must be modified to prevent vaporizing these metals. On the other hand, if metals analysis is done on an "as received" basis, a separate aliquot can be dried to determine moisture content and the metals concentration mathematically adjusted to a dry basis.
6. An equivalent pollutant (mercury, HCl) determinative or analytical procedure means a published VCS or EPA method that clearly states that the standard, practice, or method is appropriate for the pollutant and the fuel matrix and has a published detection limit equal or lower than the methods listed in Table 6 to this subpart for the same purpose.

5.4 DATA ANALYSIS

Non-Detect Data

Non-Detect data of an individual HAP will be treated as zero if all the samples result in a non-detect measurement. Otherwise, the non-detect data for the individual HAP will be treated as one-half of the method detection limit.

Statistical Outliers

In order to ensure that the data is representative of the typical fuel fired, the facility will

use the Dixon's Extreme Value[14] to determine if there is at least one outlier present in the data set.

5.5 QUALITY ASSURANCE

All samples will be handled using a chain of custody form to ensure proper procedures are followed when collecting, storing, and changing possession of samples. Hold time for mercury analysis is 28-days for liquid samples and shall not be exceeded.

The laboratory being used for fuel analysis, CAS, will perform the following quality assurance checks for every batch of samples (20 samples maximum per batch): a method bank, a matrix spike, and a matrix spike duplicate.

REVISIONS

Date	Revision	Reviser
1/18/17	Section 4.1: Added language to coal sampling from the pile would occur in the hours prior to or during the performance test.	P. LaFleur
6/1/17	Section 4.1: Added paragraph for analysis of non-performance test fuels.	
9/1/17	Section 1.0: Clarified fuel sampling requirements for performance testing (initial and new fuel type).	
	Section 2.0: Clarified fuel sampling requirements for performance testing (initial and new fuel type). Added sentence for "Samples collected from a pile…."	
	Section 4.1: Per rule revisions, added language stating a minimum of one composite sample is required for monthly fuel analysis.	
	Section 4.2: Added 63.7515(e) requirements for sampling and analysis intervals and frequencies.	
	Appendix A: Updated language of 63.7521(c) as per rule revisions.	

[14] EPA's Quality Guidance for Data Quality Assessment Practical Methods for Data Analysis EPA QA/G-9 QA00 UPDATE, July 2000

APPENDIX A SAMPLING AND SAMPLE PREPARATION PROCEDURES

40 C.F.R. § 63.7521(c)

At a minimum, for demonstrating initial compliance, you must obtain three composite fuel samples for each fuel type according to the procedures in paragraph (c) (1) or (2) of this section. For monthly fuel analyses, at a minimum, you must obtain a single composite sample. For fuel analyses as part of a performance stack test, as specified in §63.7510(a), you must obtain a composite fuel sample during each performance test run.

(1) If sampling from a belt (or screw) feeder, collect fuel samples according to paragraphs (c)(1)(i) and (ii) of this section.
   (i) Stop the belt and withdraw a 6-inch wide sample from the full cross-section of the stopped belt to obtain a minimum two pounds of sample. Collect all the material (fines and course) in the full cross-section. Transfer the sample to a clean plastic bag.
   (ii) Each composite sample will consist of a minimum of three samples collected at approximately equal intervals during the testing period.

   Note: Stopping of belts for sludge and biomass sampling cannot occur during the actual performance test runs, because it interrupts the operation of the boiler. The boiler would no longer be operating under "normal operating conditions". Sampling must occur between stack test runs. Individual stack test runs can take longer than 1 hour to complete. In addition, the sampling points must be locked-out for safety which also takes time to complete. For these reasons, individual sample collection for composite during the performance test cannot and will not practically occur at one-hour intervals. Sampling will be completed before the performance test runs, the boiler will be stabilized after the fuel collection interruption, and then the next performance test run will start.

(2) If sampling from a fuel pile or truck, collect fuel samples according to paragraphs (c) (2) (I) through (iii) of this section.
   (i) For each composite sample, select a minimum of five sampling locations uniformly spaced over the surface of the pile.
   (ii) At each sampling site, dig into the pile to a depth of 18 inches. Insert a clean shovel into the hole and withdraw a sample, making sure that large pieces do not fall off during sampling; use the same shovel to collect all samples
   (iii)Transfer all samples to a clean plastic bag for further processing.

40 C.F.R. § 63.7521(d)

Prepare each composite sample according to the procedures in paragraphs (d) (1) through (7) of this section.

(1) Thoroughly mix and pour the entire composite sample over a clean plastic sheet.
(2) Break sample pieces larger than 3 inches into smaller sizes.
(3) Make a pie shape with the entire composite sample and subdivide it into four equal parts.
(4) Separate one of the quarter samples as the first subset.

(5) If this subset is too large for grinding, repeat the procedure in paragraph (d) (3) of this section with the quarter sample and obtain a one-quarter subset from this sample.
(6) Grind the sample in a mill.
(7) Use the procedure in paragraph (d) (3) of this section to obtain a one-quarter subsample for analysis. If the quarter sample is too large, subdivide it further using the same procedure.

APPENDIX B ANALYTICAL LABORATORY CERTIFICATION

(attached if applicable)