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AIR QUALITY DIVISION

## Relative Accuracy Test Audit Test Report

Billerud Escanaba, LLC Escanaba Mill Lime Kiln Stack Escanaba, Michigan July 25, 2023

Report Submittal Date September 8, 2023

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Project No. M231813A

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## TABLE OF CONTENTS

1.0 EXECUTIVE SUMMARY	1
2.0 TEST METHODOLOGY	
Method 3A Oxygen (O <sub>2</sub> ) Determination	
3.0 TEST RESULT SUMMARY	4
4.0 CERTIFICATION	6
APPENDICES	
Appendix A - Test Section Diagram	8
Appendix B - Sample Train Diagrams	10
Appendix C - Calculation Nomenclature and Formulas	13
Appendix D - Reference Method Test Data (Computerized Sheets)	17
Appendix E - Continuous Emissions Monitoring System Data and Plant Operating Data	21
Appendix F - Calibration and Response Time Data	
Appendix G - Calibration Gas Cylinder Data	

#### 1.0 EXECUTIVE SUMMARY

Mostardi Platt conducted a Relative Accuracy Test Audit test program for Billerud Escanaba, LLC on the Lime Kiln at the Escanaba, Michigan facility. This report summarizes the results of the test program and test methods used.

The test location, test date, and test parameters are summarized below.

	TEST INFORMATION				
Test Location	Test Date	Test Parameters			
Lime Kiln	July 25, 2023	Total Reduced Sulfur (TRS) as Sulfur Dioxide (SO <sub>2</sub> )			
		Oxygen (O <sub>2</sub> )			

The purpose of the test program was to determine the relative accuracies of the Lime Kiln TRS and  $O_2$  analyzer during the specified operating condition. A complete summary of test results follows the narrative portion of this report.

Parameter	Average Reference Method Data	Average CEMS Data	Relative Accuracy Acceptance Criteria	Relative Accuracy (RA)	
TRS as SO <sub>2</sub>	5.703 ppmvd @ 10% O <sub>2</sub>	4.812 ppmvd @ 10% O <sub>2</sub>	≤ 10% of the applicable standard of 20 ppmvd @ 10% O₂	5.81% of the APS	
O <sub>2</sub>	4.583%	4.781%	≤ 1% mean difference	0.198% mean difference	

The test results from this test program indicate that the CEM system meets the United States Environmental Protection Agency (USEPA) performance specification for relative accuracy requirements as published in 40 CFR Part 60.

The gas cylinders used to perform the RATA are summarized below.

GAS CYLINDER INFORMATION						
Parameter	Gas Vendor	Cylinder Serial Number	Cylinder Value	Expiration Date		
SO <sub>2</sub>	Airgas	XC026184B	0.0 ppm	5/16/2031		
SO <sub>2</sub>	Airgas	CC506130	24.72 ppm	6/17/2026		
SO <sub>2</sub>	Airgas	CC507316	50.53 ppm	3/14/2031		
O <sub>2</sub>	Airgas	NA	0.0 %	NA		
O <sub>2</sub>	Airgas	XC026184B	5.012 %	9/27/2030		
O <sub>2</sub>	Airgas	EB0046013	10.23%	5/16/2031		
H <sub>2</sub> S	Airgas	EB0032854	30.21 ppm	9/26/2025		

The identifications of the individuals associated with the test program are summarized below.

TEST PERSONNEL INFORMATION						
Location	Address	Contact  Amanda Freele Environmental Engineer (906) 233-2603 (phone) Amanda.Freele@Billerud.com				
Test Facility	Billerud Escanaba, LLC 7100 County Road 426 M.5 Rd Escanaba, MI 49829					
Testing Company Personnel	Mostardi Platt 888 Industrial Drive Elmhurst, Illinois 60126	Christopher Jensen Senior Project Manager (630) 993-2100 (phone) cjensen@mp-mail.com Dakota Jackson Project Technician Jason Carsello Test Engineer				

#### 2.0 TEST METHODOLOGY

Emission testing was conducted following the USEPA methods specified in 40CFR60, Appendix A, in addition to the Mostardi Platt Quality Manual. Schematics of the test section diagram and sampling trains used are included in Appendix A and B respectively. Calculation nomenclature are included in Appendix C. Copies of analyzer print-outs for each test run are included in Appendix D. CEM data and process data as provided by Billerud Escanaba, LLC are also included in Appendix E.

Parameter	USEPA Reference Method	Notes/Remarks		
O <sub>2</sub> %	USEPA Method 3A, 40CFR60, Appendix A	Instrument Analysis of O <sub>2</sub> % on a dry basis		
TRS as SO <sub>2</sub>	USEPA Method 16C, 40CFR60, Appendix A	Instrument Analysis of TRS as SO <sub>2</sub> ppmvd to calculate TRS as SO <sub>2</sub> at 10 % O <sub>2</sub>		

The following test methodologies were used:

## Method 16C Total Reduced Sulfur (TRS) as Sulfur Dioxide (SO<sub>2</sub>)

Integrated gas samples were extracted from the gas stream in accordance with Method 16C (analyzer technique), 40CFR60, for the determination of total reduced sulfur (TRS) emissions from stationary sources. This method selectively removed sulfur dioxide (SO<sub>2</sub>) by bubbling the gas sample through a citrate buffer solution, then thermally oxidizing TRS compounds present to SO<sub>2</sub> via furnace tube that was set at 1100 degrees Celsius +/- 50 degrees Celsius. The gas was then sent to the THERMO Model 43i SO<sub>2</sub> analyzer. The instrument operated in the nominal range of 0 ppm to 100 ppm with the specific range determined by the high-level span calibration gas.

In addition to a zero-air calibration gas, two calibration gases were used to perform the calibration error prior to the start of the sample runs. After each run, a drift check was performed with a zero gas and the mid-level  $SO_2$  calibration gas.

In accordance with Method 16C, a performance check of the system was conducted prior to the to the start of the sample runs using a separate H<sub>2</sub>S gas. System performance checks were performed after every third test run and used to correct the TRS data.

The Model 43i operates on the principle that SO<sub>2</sub> molecules absorb ultraviolet (UV) light and become excited at one wavelength, then decay to a lower energy state emitting UV light at a different wavelength. Specifically,

$$SO_2 + hv_1 \rightarrow SO_2^* \rightarrow SO_2 + hv_2$$

The sample is drawn into the Model 43*i* through the sample bulkhead. The sample flows through a hydrocarbon "kicker", which removes hydrocarbons from the sample by forcing the hydrocarbon molecules to permeate through the tube wall. The SO<sub>2</sub> molecules pass through the hydrocarbon "kicker" unaffected.

The sample flows into the fluorescence chamber, where pulsating UV light excites the  $SO_2$  molecules. The condensing lens focuses the pulsating UV light into the mirror assembly. The mirror assembly contains four selective mirrors that reflect only the wavelengths which excite  $SO_2$  molecules.

As the excited  $SO_2$  molecules decay to lower energy states, they emit UV light that is proportional to the  $SO_2$  concentration. The bandpass filter allows only the wavelengths emitted by the excited  $SO_2$  molecules to reach the photomultiplier tube (PMT). The PMT detects the UV light emission from the decaying  $SO_2$  molecules. The photodetector, located at the back of the fluorescence chamber, continuously monitors the pulsating UV light source and is connected to a circuit that compensates for fluctuations in the lamp intensity.

As the sample leaves the optical chamber, it passes through a flow sensor, a capillary, and the "shell" side of the hydrocarbon kicker. The Model 43*i* outputs the SO<sub>2</sub> concentration to the front panel display, the analog outputs, and also makes the data available over the serial or Ethernet connection.

Stack gas was delivered to the analyzer through a heated extractive gas sampling system. The glass probe and Teflon filter area were heated to a minimum of 250 degrees Fahrenheit. The entire system was calibrated in accordance with the Method.

### Method 3A Oxygen (O<sub>2</sub>) Determination

Stack gas  $O_2$  concentrations were determined in accordance with USEPA Method 3A. A Servomex analyzer was used to determine the  $O_2$  concentrations in the manner specified in the Method. The instrument has a paramagnetic detector and the  $O_2$  operates in the nominal range of 0% to 25% with the specific range determined by the high-level calibration gas. High-range calibrations were performed using USEPA Protocol gas. Zero nitrogen (a low ppm pollutant in balance nitrogen calibration gases) was introduced during other instrument calibrations to check instrument zero. High- and a mid-range %  $O_2$  levels in balance nitrogen were also introduced. Zero and mid-range calibrations were performed using USEPA Protocol gas after each test run. Copies of the gas cylinder certifications are found in Appendix G. This testing met the performance specifications as outlined in the Method.

### 3.0 TEST RESULT SUMMARY

Client: Billerud Escanaba, LLC

Location: Lime Kiln

Facility: Escanaba Mill

Date: 7/25/23

Project #: M231813

Test Method: 16C, 3A

Applicable Standard: 20

#### TRS as SO2 RATA

**Primary CEM Monitor Information** 

so	<sub>2</sub> Moni	tor/Model:	TECO 4	3iQ-CAN		SO <sub>2</sub> Serial #: 12218618488		618488
0	<sub>2</sub> Moni	tor/Model:	TECO	Citicel	O <sub>2</sub> Serial # :		CC031506-3	
1=accept 0=reject	Test Run	Test Date	Start Time	End Time	RM SO2 ppmvd @ 10 % O2	CEM SO2 ppmvd @ 10 % O2	(RM-CEM) Difference (di)	(RM-CEM) Difference <sup>2</sup> (di <sup>2</sup> )
1	1	07/25/23	08:00	08:29	6.60	5.88	0.72	0.518
1	2	07/25/23	08:45	09:14	7.08	6.93	0.15	0.023
1	3	07/25/23	09:30	09:59	5.80	4.90	0.90	0.810
1	4	07/25/23	10:39	11:08	7.15	6.21	0.94	0.884
1	5	07/25/23	11:27	11:56	5.76	4.80	0.96	0.922
0	6	07/25/23	12:11	12:40	4.45	3.66	0.79	0.624
1	7	07/25/23	15:28	15:57	5.75	4.57	1.18	1.392
1	8	07/25/23	16:11	16:40	4.66	3.57	1.09	1.188
1	9	07/25/23	16:55	17:24	3.58	2.89	0.69	0.476
1	10	07/25/23	17:59	18:28	4.95	3.56	1.39	1.932
				t(0.975)		9		
		Mean Re	ference Me	thod Value	5.7	703	RM avg	
			Mean	<b>CEM Value</b>	4.812		CEM avg	
			Sum of	Differences	8.020		di	
Mean Difference			0.891		d			
Sum of Differences Squared		8.145		di <sup>2</sup>				
Standard Deviation		0.3	353	sd				
(	Confide	ence Coeff	icient 2.5% l	Error (1-tail)	0.272		cc	
Relative Accuracy - APS			5.	81	RA			

A Relative accuracy based upon +/- 10% of applicable standard

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Client: Billerud Escanaba, LLC

Location: Lime Kiln Date: 7/25/23

Facility: Escanaba Mill Project #: M231813

Test Method: 3A

O<sub>2</sub> % (dry) RATA

Primary CEM Monitor Information

O <sub>2</sub>	Monit	or/Model:	TECO	Citicel		O <sub>2</sub> Serial # :	CC03	1506-3
1=accept 0=reject	Test Run	Test Date	Start Time	End Time	RM O <sub>2</sub> % (dry)	CEM O <sub>2</sub> % (dry)	(RM-CEM) Difference (di)	(RM-CEM) Difference <sup>2</sup> (di <sup>2</sup> )
1	1	07/25/23	08:00	08:29	4.25	4.06	0.19	0.04
1	2	07/25/23	08:45	09:14	5.50	5.00	0.50	0.25
1	3	07/25/23	09:30	09:59	4.47	4.30	0.17	0.03
1	4	07/25/23	10:39	11:08	4.61	5.42	-0.81	0.66
1	5	07/25/23	11:27	11:56	4.44	5.52	-1.08	1.17
0	6	07/25/23	12:11	12:40	6.92	4.48	2.44	5.95
1	7	07/25/23	15:28	15:57	4.31	4.10	0.21	0.04
1	8	07/25/23	16:11	16:40	4.49	4.88	-0.39	0.15
1	9	07/25/23	16:55	17:24	4.65	4.42	0.23	0.05
1	10	07/25/23	17:59	18:28	4.53	5.33	-0.80	0.64
		M D.	f M.	t(0.975)	2.3	9 306	DM	
		Mean Re		thod Value CEM Value		583 781	RM avg	
				Differences	-1.780 di			
Mean Difference			-0.198		d			
Sum of Differences Squared			3.	3.027		di <sup>2</sup>		
	Standard Deviation		0.	0.578		sd		
C	onfide	nce Coeff	icient 2.5% l	Error (1-tail)	0.444 cc		cc	
			Relativ	e Accuracy	0.	198	RA	

### 4.0 CERTIFICATION

Mostardi Platt is pleased to have been of service to Billerud Escanaba, LLC. If you have any questions regarding this test report, please do not hesitate to contact us at 630-993-2100.

As program manager, I hereby certify that this test report represents a true and accurate summary of emissions test results and the methodologies employed to obtain those results, and the test program was performed in accordance with the methods specified in this test report.

MOSTARDI PLATT

Program Manager

Quality Assurance

## **APPENDICES**

Appendix A - Test Section Diagram

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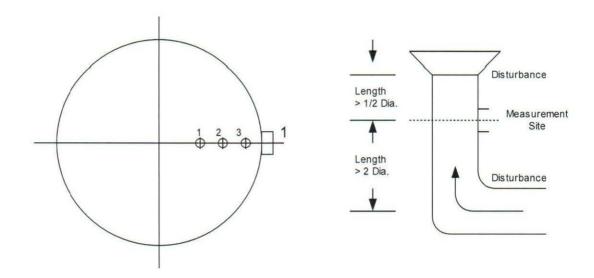
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Page 8 of 53

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#### **GASEOUS TRAVERSE FOR ROUND DUCTS**



Job: Billerud Escanaba, LLC

Escanaba Mill

Escanaba, Michigan

Date: July 25, 2023

Test Location: Lime Kiln

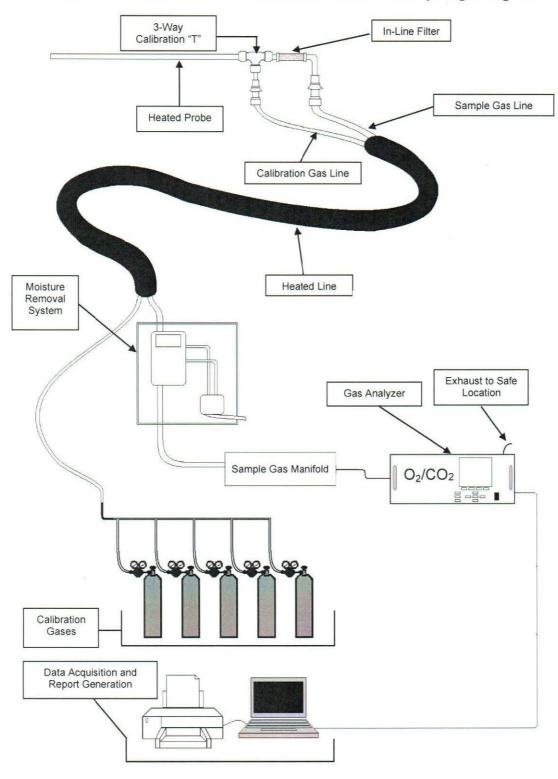
Duct Diameter: 4.667 feet

Duct Area: 17.11 square feet

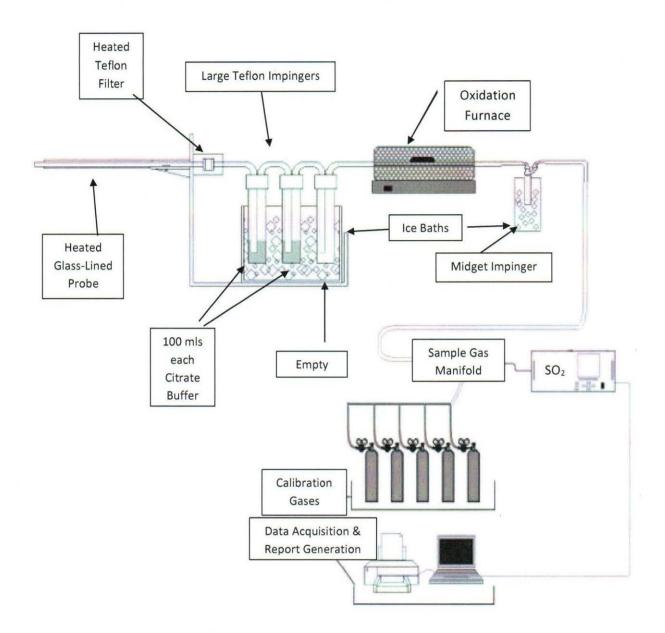
No. Sample Points: 3

## Appendix B - Sample Train Diagram

## **USEPA Method 3A Extractive Gaseous Sampling Diagram**



## USEPA Method 16C - Total Reduced Sulfur Sample Train Diagram



## Appendix C - Calculation Nomenclature and Formulas