1. Introduction

1.1 Summary of Test Program

AECOM Technical Services Inc. (AECOM) was contracted by DDP Specialty Electronic Materials US, Inc. (DuPont) in Midland, Michigan, Specialty Monomers (Spec Mono) Plant to conduct performance testing on their site Tar Incinerator (EU95) during the week of June 15, 2020. The performance testing consisted of measurements for nitrogen oxides (NO_x). The following sections present the regulatory background, objectives, description, and schedule of the planned testing program.

Table 7 of the CISWI Guidelines Rule (40 CFR 60 Subpart DDDD) requires that observations for fugitive ash be conducted during performance testing, however, the incinerator does not create ash or have an ash handling system. DDP/DuPont proposed using the installed Continuous Opacity Monitor (COMs) currently installed on the unit to demonstrate compliance with this requirement of the rule. EGLE waived the fugitive ash observations and approved use of the COMs to demonstrate compliance with the opacity limit in the test plan approval letter dated 5/28/20.

The results of testing are presented in Table 1-1. Details supporting these data are presented in the balance of this report.

Table 1-1 Emission Testing Results

Sample Type	Test Method	Sampling Time (min/run)	Allowable Emission Rate	Actual Emission Rate
NO_x	EPA Method 7E	60 min	76 ppmv @ 7% O_2	49.15 ppmv @ 7% O_2

1.2 Regulatory Background

On March 21, 2011, in parallel with the publication of the Boiler National Emission Standard for Hazardous Air Pollutants (NESHAP) rules and the Non-Hazardous Secondary Material (NHSM) rule, EPA promulgated the final updates to the New Source Performance Standards (NSPS) and Emission Guidelines (EG) for Existing CISWI Units, collectively referred to as the "2011 CISWI Rules." The 2011 CISWI Rules impact any facility that owns an emission unit that "combusts, or has combusted in the preceding six months, any solid waste as that term is defined in 40 CFR Part 241.2." The CISWI rules were then reconsidered and amended in 2013. The final version of the CISWI Rules/Guidelines were published in the Federal Register on February 7, 2013. The final rule is titled: Subpart DDDD— Emissions Guidelines and Compliance Times for Commercial and Industrial Solid Waste Incineration Units. EGLE then promulgated the state rules on January 2, 2019: Rule R 336.1974 Emissions Standards for Existing Commercial and Industrial Solid Waste Incinerators.

Per Section 60.2720(a)(3) of the Federal CISWI rule as referenced by the Michigan Rule R 336.1974(9)(g), DDP is reducing the frequency of testing for certain constituents to a triennial frequency following successful completion of the previous two tests. Successful completion of the test were test results at a level equal to or less than 75% of the applicable emission limit in both the July 2018 and June 2019 performance tests. Parameters measured during the June 2020 performance test include nitrogen oxides (NO_x). O_2 concentrations were also measured for excess air correction.

This test protocol addresses the compliance test.

Responsible Groups	• DDP Specialty Electronic Materials US, Inc. (DuPont) • Michigan Department of Environment, Great Lakes, and Energy (EGLE) • Environmental Protection Agency (EPA)
Applicable Regulations	• MI-ROP-A4033-2017b • 40 CFR Part 60 Commercial and Industrial Solid Waste Incineration Units MACT (Subpart DDDD) • EGLE Air Quality Division Part 9, Rule 336.1974"
Industry/Plant	• Specialty Monomers, 1130 Building
Plant Location	• Midland, Michigan I-Park Facilities, 48640
Unit Initial Start-up	• 1990
Air Pollution Control Equipment	• N/A
Emission Points	• EU-95 Tar Incinerator (EU95-S1)
Pollutants/Diluent Measure	• Nitrogen Oxides (NO_x) • Oxygen (O_2)
Test Dates	• June 16, 2020

1.3   Key Personnel

The key personnel coordinating this test program are:

- Alex Gagliardi provided support as the Process Focal Point. The Process Focal Point is responsible for coordinating the plant operation during the test and ensuring the unit is operating at the agreed-upon conditions in the test plan. They also serve as the key contact for collecting any process data required and providing all technical support related to process operation.

- Teresa Schwartz provided support as the Environmental Focal Point for this test. The Environmental Focal Point is responsible for ensuring that all regulatory requirements and citations are reviewed and considered for the testing. All agency communication will be completed through this role. Contact information is 989-264-7105.

- James Edmister served as the Test Plan Coordinator. The Test Plan Coordinator is responsible for the overall leadership of the sampling program. They also develop the overall testing plan and determine the correct sample methods.

- Daniel J Nuñez provided support as a technical review of the test data.

- James Edmister served as the Sample Team Leader. The sample Team Leader is responsible for ensuring the data generated meets the quality assurance objectives of the plan. Randy Reinke assisted as the sampling technician.

2. Plant and Sampling Location Descriptions

2.1 Facility Description

DuPont operates a tar incinerator (EU95-S1) at its Midland, Michigan chemical manufacturing facility. EU95-S1 is a boiler that produces steam from the heat input of natural gas and process tars. The process tars contain distillation heavies from the 1130 building process and process aids from the distillation process. The boiler is rated for 48 MMBtu/hr while the burner is rated for 15 MMBtu/hr. EU95-S1 must meet the requirements of the Commercial and Industrial Solid Waste Incineration (CISWI) rule promulgated under 40 CFR Part 60 Subpart DDDD, as referenced by EGLE Rule R 336.1974, and is regulated as an Energy Recovery Unit under the rule.

2.2 Performance Test Operations

The Performance Test was conducted at one operating condition to demonstrate the system performance with respect to the emission standards listed in this test report. During each test run, CMS parameters were monitored, and stack gas emissions were measured. The following sections briefly summarize these activities associated with the Performance test.

2.2.1 Unit Process Data

Process monitoring information pertinent to establishing that the unit is operating at normal conditions was recorded during the test by the EU-95 Tar Incinerator data acquisition system. One-minute average data was obtained from the process control system for each operating parameter specified in the test plan for each test run. For each operating parameter, an average value was calculated for each test run.
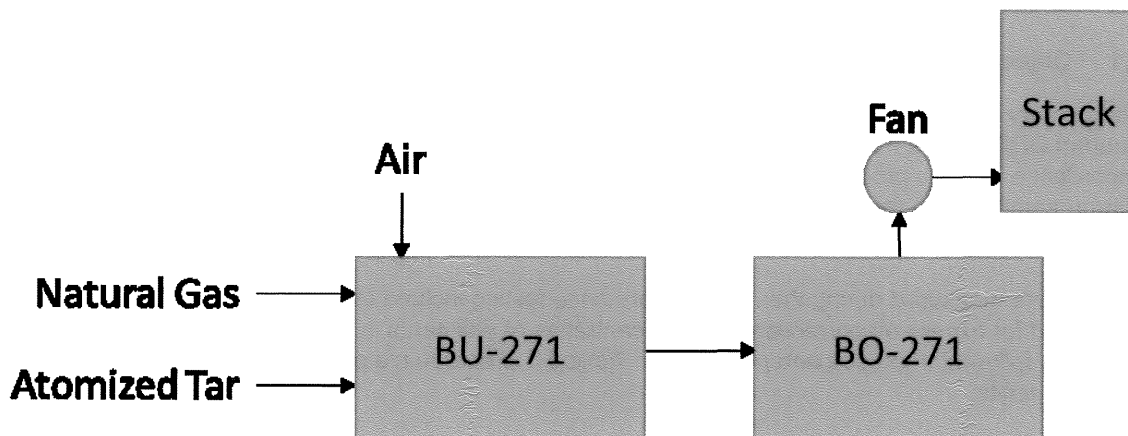
Figure 1 EU95 Tar Incinerator Process Schematic

Table 2-1 Manufacturer's Name and Model Number

Equipment	Manufacturer	Model Number
BU-271	Bloom	S-1610-022
BO-271	Johnston	509 Series

3. Summary and Discussion of Test Plan

3.1 Objectives and Test Matrix

The primary objective of this testing is to demonstrate compliance with the requirements of 40 CFR 60 Subpart DDDD. The Performance Testing of the Incinerator Stack NO_x emissions was performed in strict accordance with the procedures specified in 40 CFR 60, Appendix A. This test report describes the instrumental procedures performed on the Incinerator Stack located within the DuPont Specialty Monomers Plant.

Parameters measured during the June Performance testing include NO_x. O_2 concentrations were also measured for excess air correction. The concentration of pollutants in the exhaust gas were measured using the following methods and procedures: Table 3-1 presents the parameters test methods and the emission limits.

Table 3-1 Test Matrix and Objectives

Parameter	Test Method	Regulation	Emission Limit
O_2/CO_2	EPA Method 3A	40 CFR 60, Subpart DDDD	N/A
NO_x	EPA Method 7E	40 CFR 60, Subpart DDDD	76 ppmv @ 7% O_2

The compliance test was conducted on June 16, 2020 under normal process operating conditions. The emission testing of the Incinerator Stack consisted of three (3) test runs for NO_x.

3.2 Process Operating Rates

As required by the regulation and EGLE guidance, all sampling was completed at normal operating conditions. The table below outlines the proposed operating rates.

The normal operating rates were determined by reviewing the process data from the previous six months of operation and deciding the typical operating range of the unit. The average values do not include calibration data, startup date, shutdown data, malfunction data, and data obtained not burning waste.

Parameter	Normal Operating Rate	Operating Rate During Testing
Heat Input (MMBtu/hr)	4-13	8-9
Tars Feed Rate (lb/hr)	180-420	400-425
Natural Gas Feed Rate (scfh)	1450-9000	1420-1510
O_2 in Vent Stack (%)	9-15	10-11

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Table 3-2 Testing Run Data (NO_x)

	Run 1	Run 2	Run 3	Average
Run Date	6/16/2020	6/16/2020	6/16/2020	
Run Times	09:30-10:30	10:50-11:50	12:08-13:08	
Stack Gas O_2 (%)	12.21	12.24	12.28	12.24
Nitrogen Oxides				
ppmdv	29.9	30.8	31.1	30.6
ppmdv @7% O_2	47.86	49.41	50.19	49.15

4. Sampling and Analytical Procedures

4.1 Sample Time

The duration of each test run for instrumental method (NO_x) was sixty (60) minutes.

4.2 Sample Test Runs

Three (3) sample test runs were performed for each method.

4.3 Sample Port Location

The stack is approximately 40-ft high with an inside diameter of 35 inches at the elevation of the sampling points. The sampling ports are approximately 64 -in downstream from the closest disturbance (stack breach) and 108 -in upstream from the next nearest disturbance (stack exit). The number of sampling points at this port location will be determined in accordance with EPA Method 1. Figure 2 and Figure 3 present schematics of the sampling points and location.

4.4 Instrumental Methods

Emission gas was withdrawn from the Incinerator Stack and transported to the AECOM CEMS located at ground level. A stainless-steel sampling probe was inserted into the stack and used to collect sample gas. A heated Teflon sample line was used to transport the sample gas from the sampling probe to the CEMS. At the mobile laboratory, stack exhaust gas was dried using a condenser and routed to the individual analyzers for analysis on a dry basis. Data were collected using a dedicated data acquisition system. The system stores the data as fifteen-second averages.

Each analyzer was calibrated before testing using gas standards as specified by EPA Methods 7E and 3A. Only EPA Protocol gases or certified pure zero nitrogen and air gases were used for calibration.

Method compliance is ensured by performing:

- Calibration error (challenging the calibrated instrument at three levels)
- System drift (challenging the overall system at two levels)
- System response testing
- Stratification check demonstrating lack of stratification and allowing sample gas to be collected from a single point.
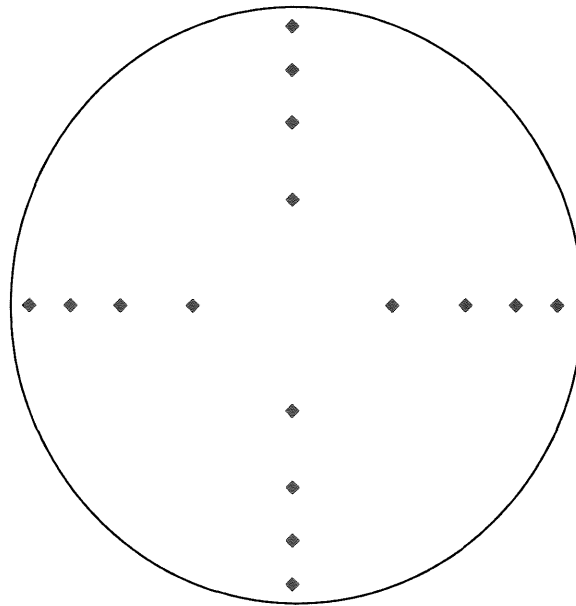- Calibration drift (repeating system bias after testing)

A schematic of the instrumental sampling system is shown in Figure 4.

The following instruments were used:

- EPA Method 3A (O_2/CO_2) - Teledyne model 300M; paramagnetic
- EPA Method 7E (NO_x) - TECO Model 42; chemiluminescent NO detector.

Figure 2 Sample Points

Stack Diameter 2.9 feet

Point	Percentage of Diameter	Distance from Wall (feet)
1	3.2	0.09
2	10.5	0.31
3	19.4	0.57
4	32.3	0.94
5	67.7	1.97
6	80.6	2.35
7	89.5	2.61
8	96.8	2.82

Figure 3 Sample Location

Stack Exit

Downstream Disturbance
Stack exit

Distance A
108 inches

Port

Port

Distance B
64 inches

Flow

Upstream Disturbance
Stack inlet

Inlet
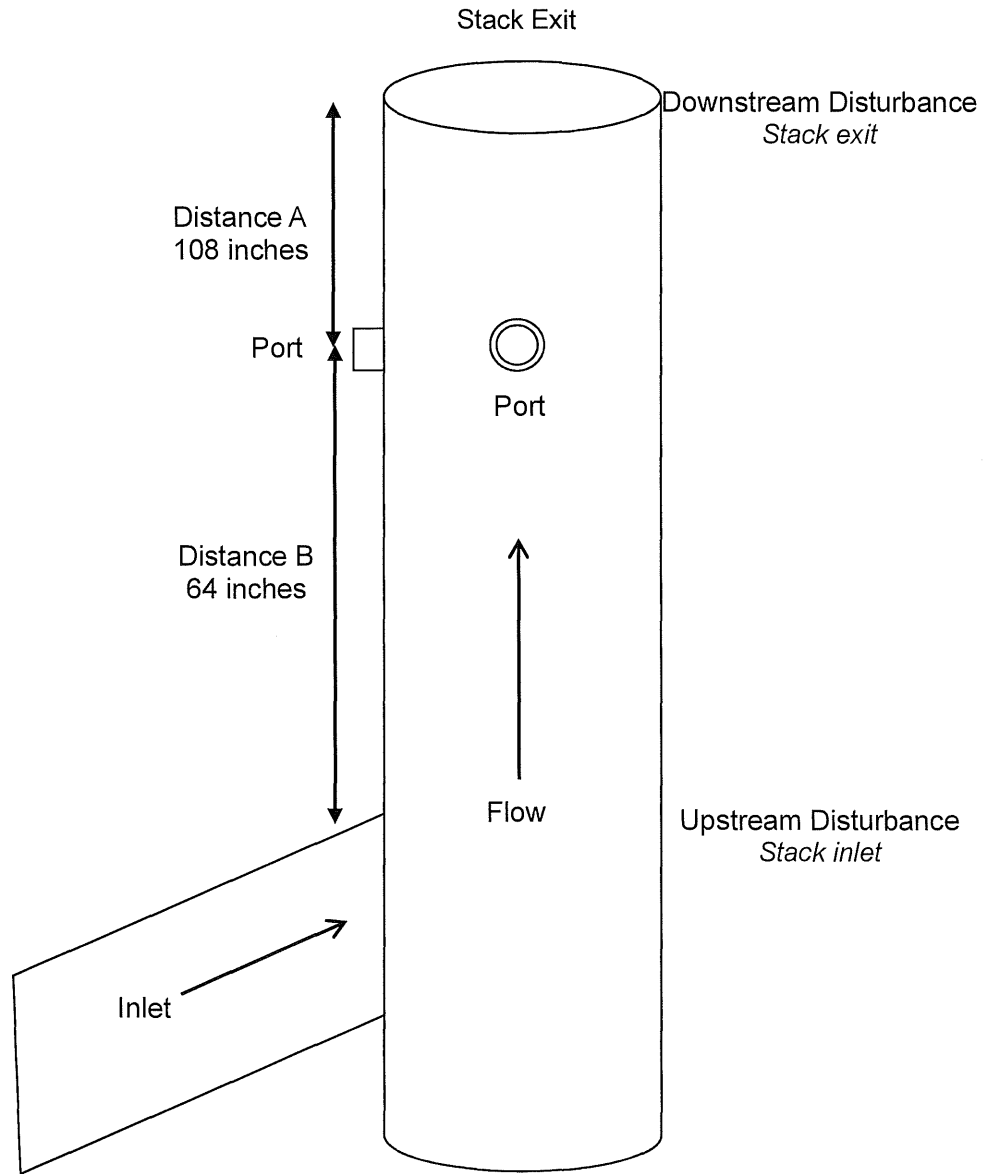
Figure 4 Schematic of AECOM CEM System
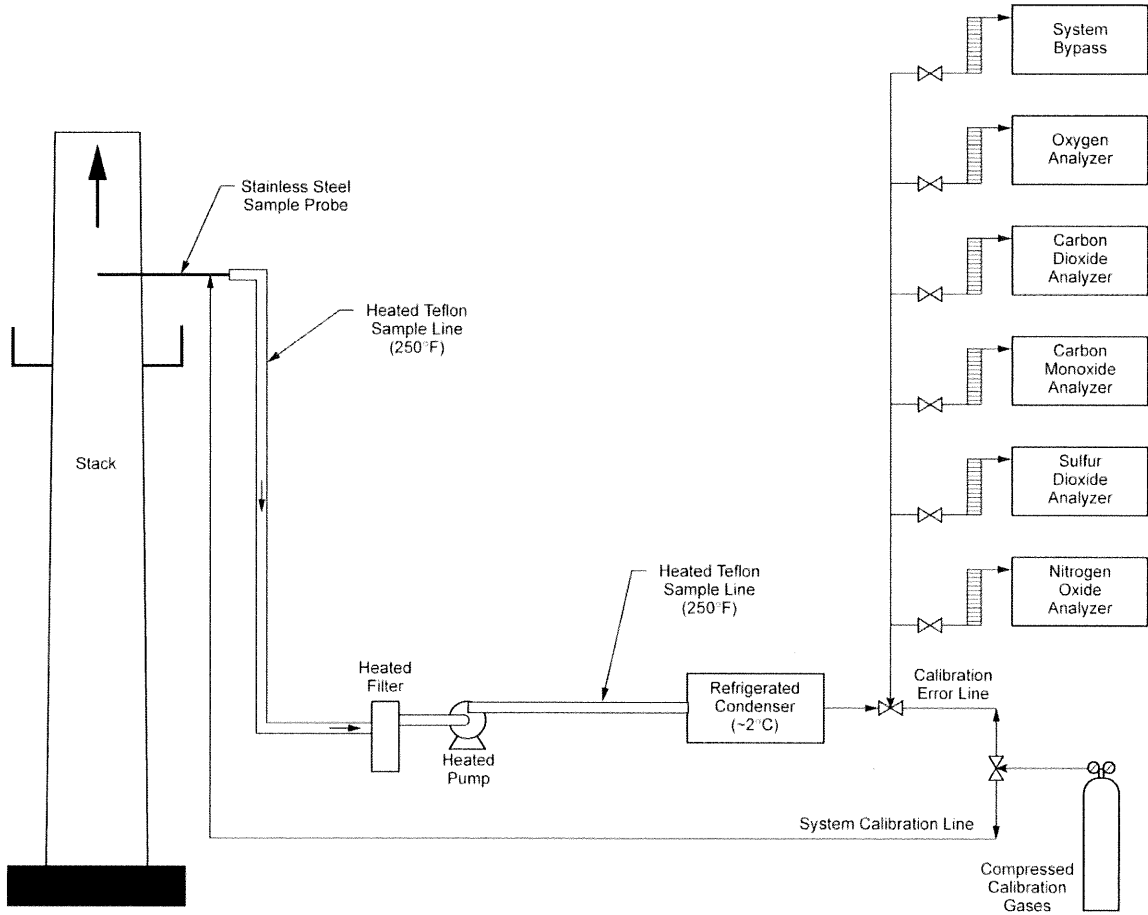
5. Calculation Examples

Analyzer Calibration Error Calculations

The calibration error test consisted of challenging each reference monitor at three measurement points against known calibration gas values. Calibration error for the reference is calculated using the following equation:

$$CE_{RM} = \frac{|Analyzer\ Response - Calibration\ Gas\ Value|}{Span\ of\ Analzyer} \times 100$$

Reference O_2 Calibration Error Example (Run 1)

$$CE_{RM} = \frac{|(0.1\ \%) - (0.00\ \%)|}{(19.71\ \%)} \times 100 = \underline{0.0\ \%}$$

$$CE_{RM} = \frac{|(19.73\ \%) - (19.71\ \%)|}{(19.71\ \%)} \times 100 = \underline{0.1\ \%}$$

$$CE_{RM} = \frac{|(9.89\ \%) - (9.98\ \%)|}{(19.71\ \%)} \times 100 = \underline{0.5\ \%}$$

System Calibration Bias Calculations

The system bias calibration test consisted of challenging the reference sample system at two measurement points against the local calibration values. Calibration bias calculations for the reference sample system are calculated using the following equation:

$$CB_{RM} = \frac{System\ Calibration\ Response - Analzyer\ Calibration\ Response}{Span\ of\ Analzyer} \times 100$$

Reference O$_2$ Initial System Bias Example (Run 1)

$$CB_{RM} = \frac{|(0.01\%) - (0.10\ \%)|}{(19.71\ \%)} \times 100 = \underline{0.5\ \%}$$

$$CB_{RM} = \frac{|(9.89\ \%) - (9.85\ \%)|}{(19.71\ \%)} \times 100 = \underline{-0.2\ \%}$$

Calibration Drift Calculations

The calibration drift tests were conducted at the beginning and end of each run. Analyzer maintenance, repair or adjustment could not be completed until the system calibration response was recorded. Calibration drift for the reference is calculated using the following equation:

$$CD_{RM} = \frac{|Final\ System\ Cal\ Response - Initial\ System\ Cal\ Response|}{Span\ of\ Analzyer} \times 100$$

Reference O₂ Calibration Drift Run #1 Example

$$CD_{RM} = \frac{|(0.10\ \%) - (0.10\ \%)|}{(19.71\ \%)} \times 100 = \underline{0.0\ \%}$$

$$CD_{RM} = \frac{|(9.85\ \%) - (9.87\ \%)|}{(19.71\ \%)} \times 100 = \underline{-0.1\ \%}$$

System Calibration Drift Correction

The gas concentrations are corrected for the system calibration bias. The concentrations are calculated using the following equations:

$$C_{Gas} = (\overline{C} - C_O)\left(\frac{C_{MA}}{C_M - C_O}\right)$$

where: C_{Gas} = Effluent Concentration, dry ppm or %
   $\overline{C}$ = Average Analyzer Concentration, ppm or %
   C_O = Average Initial and Final System Calibration
       Responses for Zero Gas, ppm or %
   C_M = Average Initial and Final System Calibration
       Responses for Upscale Calibration Gas, ppm or %
   C_{MA} = Actual Concentration of Upscale Calibration Gas, ppm or %

O₂ System Calibration Drift Correction for Run #1 Example

$$C_{Gas} = (12.04\ \% - 0.11\ \%)\left(\frac{9.98\ \%}{9.86\% - 0.11\ \%}\right) = \underline{12.21\ \%}$$

Effluent Concentration for Oxygen Correction

The gas concentrations are corrected to 7% O_2. The concentrations are calculated using the following equation:

$$P_{Corr} = P_{meas} \times \frac{20.9 - O_{2\,std}}{20.9 - O_{2\,meas}}$$

P_{Corr} = Pollutant Concentration, corrected to the oxygen standard

P_{meas} = Measured concentration of Pollutant

$O_{2\,std}$ = Oxygen concentration to be used for a standard

$O_{2\,meas}$ = Oxygen concentration measured

Effluent Concentration Corrected for Oxygen Concentration Run #1 Example

For NO_x Run 1

P_{meas}	29.92	ppm
$O_{2\,std}$	7.00	%
$O_{2\,meas}$ =	12.21	%

$$P_{Corr} = 29.92 \quad X \quad \frac{(\quad 20.90 \quad - \quad 7.00 \quad)}{(\quad 20.90 \quad - \quad 12.21 \quad)}$$

P_{Corr} = 47.86ppm

6. Field test Data