

DDP Specialty Electronic Materials US, LLC (DDP/DuPont) Michigan Operations

CISWI Compliance Testing
1130 Building Specialty Monomer,
EU95 Tar Incinerator

MI-ROP-P1027-2020b
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1. Introduction

1.1 Summary of Test Program

AECOM Technical Services, Inc. (AECOM) has been contracted by DDP Specialty Electronic Materials US, LLC (DDP/DuPont) to conduct emissions performance testing on their site Tar Incinerator (EU95) at their Specialty Monomers (Spec Mono) Plant in Midland, Michigan on June 13th, 2023. The Commercial and Industrial Solid Waste Incineration Units (CISWI) compliance performance testing consist of measurements for nitrogen oxides (NO_x) and visible emissions (VE). The following sections present the regulatory background, objectives, description, and schedule of the planned testing program.

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 | Actual Emission |
|-----------------|---------------|-------------------------|------------------------------|--------------------------------|
| NO _x | EPA Method 7E | 60 min | 76 ppmvd @ 7% O ₂ | 55.6 ppmvd @ 7% O ₂ |
| VE | EPA Method 9 | 60 min | 10% opacity | 0% opacity |

1.2 Regulatory Background

On March 21, 2011, in parallel with 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.

In accordance with the requirements of 40 CFR 60 Subpart DDDD, each affected unit must conduct an annual performance test. The requirements are outlined in 40 CFR 60.2690 and in tables 2 or 6-9, depending on the specific mechanism by which the unit is affected.

The following table summarizes the pertinent data for this compliance test:

Table 1-2. Compliance Summary

| | |
|--|---|
| Responsible Groups | <ul style="list-style-type: none"> • DDP Specialty Electronic Materials US, LLC (DDP/DuPont) • Michigan Department of Environment, Great Lakes, and Energy (EGLE) • United States Environmental Protection Agency (US EPA) |
| Applicable Regulations | <ul style="list-style-type: none"> • MI-ROP-P1027-2020b • 40 CFR 60, Subpart DDDD: Commercial and Industrial Solid Waste Incineration Units (CISWI) • "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 | • Low NO _x burner technology, low excess air firing |
| Emission Points | • EU95 Tar Incinerator (EU95) |
| Pollutants/Diluent Measure | <ul style="list-style-type: none"> • Visible Emissions (VE) • Nitrogen Oxides (NO_x) • Oxygen/Carbon Dioxide (O₂/CO₂) |
| Test Date | • June 13 th , 2023 |

1.3 Key Personnel

Names and affiliations of personnel, including their roles in the test program, are summarized in the following table (Table 1-3).

Table 1-3. Key Personnel

| Role | Role Description | Name | Affiliation |
|---------------------------|--|------------------------|--------------------|
| Process Focal Point | <ul style="list-style-type: none"> • Coordinate plant operation during the test. • Ensure the unit is operating at the agreed upon conditions in the test plan. • Collect any process data required. • Provide all technical support related to process operation. | Matt Lloyd | DuPont |
| Environmental Focal Point | <ul style="list-style-type: none"> • Ensure all regulatory requirements and citations are reviewed and considered for the testing. | Randy Reinke | DuPont |
| Technical Reviewer | <ul style="list-style-type: none"> • Completes technical review of the test data. | Christopher Trevillian | AECOM |
| Field Team Leader | <ul style="list-style-type: none"> • Ensures field sampling meets the quality assurance objectives of the plan. | Peter Becker | AECOM |
| Sample Project Leader | <ul style="list-style-type: none"> • Ensures data generated meets the quality assurance objectives of the plan. | James Edmister | AECOM |

2. Plant and Sampling Location Description

2.1 Facility Description

DuPont operates a tar incinerator (EU95) at its Midland, Michigan chemical manufacturing facility. EU95 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 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 (ERU) 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 Table 1-1. During each test run, continuous monitoring system (CMS) parameters were recorded, and stack gas emissions were measured. The following sections briefly summarize these activities associated with the Performance test.

2.3 Unit Process Data

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

Figure 2-1. Sample Train Schematic

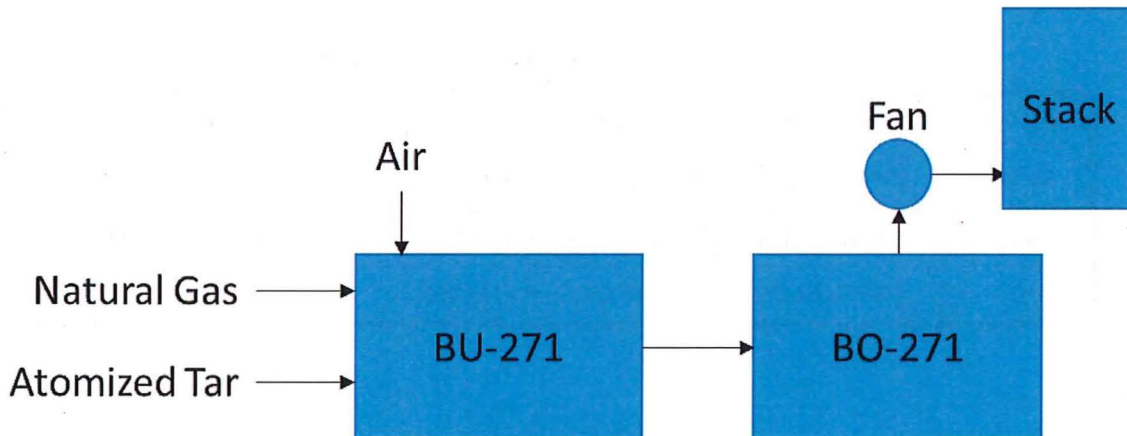


Table 2-1. Manufacturer's Name and Model Number

| 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 was to demonstrate compliance with the requirements of 40 CFR 60 Subpart DDDD. The performance testing of the incinerator stack NO_x and visible emissions was performed in accordance with the procedures specified in 40 CFR 60, Appendix A. This test report describes the procedures performed on the incinerator stack located within DuPont's Specialty Monomers Plant.

Parameters measured during the June performance testing included NO_x, VE, O₂, and CO₂. O₂ and CO₂ concentrations were measured for molecular weight and excess air correction. The concentrations of pollutants in the exhaust gas were measured by using the following methods and procedures:

- EPA Method 3A, "Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources."
- EPA Method 7E, "Determination of Nitrogen Oxides Emission from Stationary Sources.";

The emission testing of the incinerator stack consisted of three (3) test runs each for NO_x, VE, O₂, and CO₂.

The duration of each test was as followed:

- Instrumental methods (NO_x, VE, O₂, and CO₂) tests were a minimum sixty (60) minutes in duration

The applicable limits demonstrated during the compliance test as well as the methods employed are listed in **Table 3-1**.

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 ppmvd @ 7% O ₂ |
| VE | EPA Method 9 | 40 CFR 60, Subpart DDDD | 10% |

3.2 Process Operating Rates

As required by the regulation and EGLE guidance, all sampling was completed at normal operating conditions.

The normal operating rates were determined by reviewing the process data from the previous six months of operation. The average values do not include calibration data, startup data, shutdown data, malfunction data, and data obtained not burning waste. The Parameters are shown in **Table 3-2**.

Table 3-2. Process Operating Rates

| Parameter | Normal Operating Rate | Operating Rate During Testing |
|----------------------------------|-----------------------|-------------------------------|
| Heat input (MMBtu/hr) | 4-13 | 7.5 |
| Tars Feed Rate (lb/hr) | 180-420 | 359-360 |
| Natural Gas Feed Rate (scfh) | 1450-9000 | 1495-1505 |
| O ₂ in Vent Stack (%) | 9-15 | 10.7 |

The results of the compliance test are listed below in **Table 3-3**.

Table 3-3. Test Results Data (NO_x and Visual Emissions)

| | Run 1 | Run 2 | Run 3 | Average |
|------------------------------|------------|-------------|-------------|---------|
| Run Date | 06/13/2023 | 06/13/2023 | 06/13/2023 | - |
| Run Times | 9:00-10:00 | 10:15-11:15 | 11:27-12:27 | - |
| Stack Gas O ₂ (%) | 12.30 | 12.31 | 12.28 | 12.30 |
| Nitrogen Oxides | | | | |
| ppmvd | 33.8 | 34.4 | 34.9 | 34.4 |
| ppmvd @7% O ₂ | 54.7 | 55.7 | 56.3 | 55.6 |
| Visual Emissions | 0% | 0% | 0% | 0% |

4. Sampling and Analytical Procedures

4.1 Sample Time

The duration of each test run for instrumental methods NO_x was sixty (60) minutes. There are no minimum sample volume requirements for EPA methods 3A and 7E.

4.2 Sample Test Runs

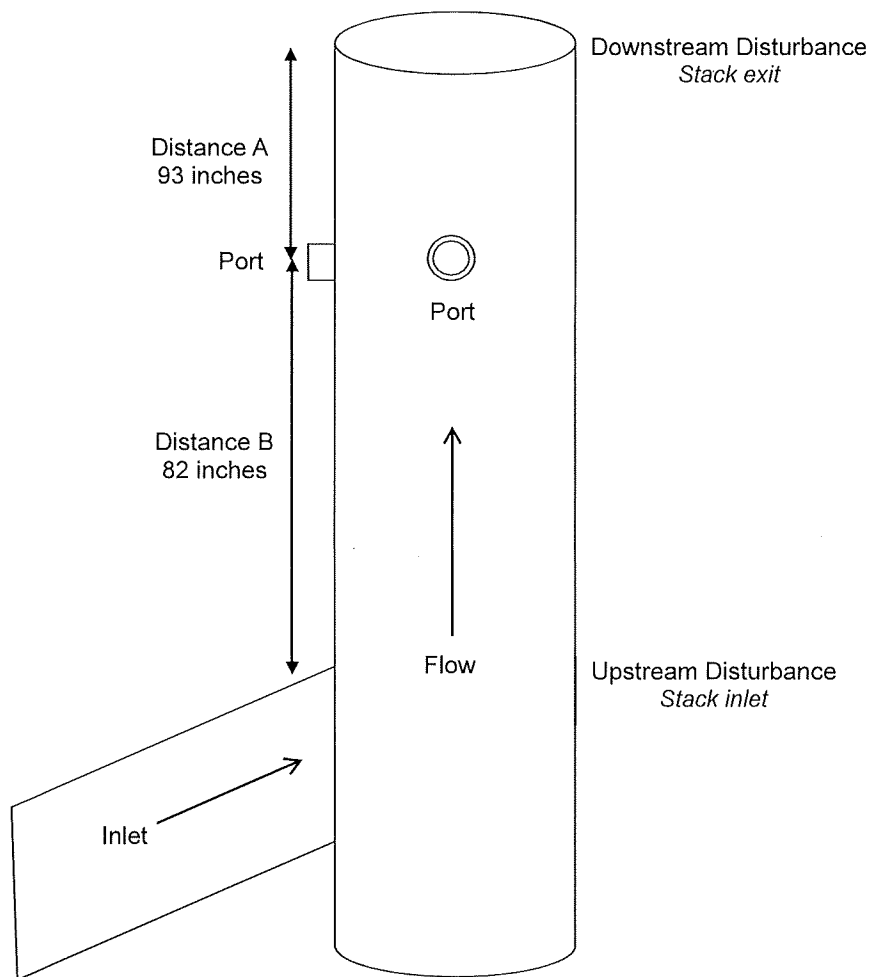
Three (3) sample test runs were performed for each method.

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 82 inches downstream from the closest disturbance (stack breach) and 93 inches upstream from the next nearest disturbance (stack exit).

Figure 4-1 illustrates the sampling location.

Figure 4-1. Sample 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 (O_2 , CO_2 , and NO_x). Data was collected using a dedicated data acquisition system. The system stores the data as ten second averages.

Each analyzer was calibrated before testing using gas standards as specified by EPA Methods 3A and 7E. 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)

Flue Gas Molecular Weight – EPA Method 3A

EPA Method 3A (Instrumental Method) was utilized to determine the diluent during each run on the outlet.

An analyzer measured O_2 content on the basis of the strong paramagnetic properties of O_2 relative to other compounds present in combustion gases. In the presence of a magnetic field, O_2 molecules become temporary magnets. The analyzer determines the sample gas O_2 concentration by detecting the displacement torque of the sample test body in the presence of a magnetic field.

Determination of Nitrogen Oxides – EPA Method 7E

EPA Method 7E was utilized to determine nitrogen oxide concentrations during each run on the outlet.

An analyzer measured NO_x using chemiluminescence technology. Ozone is combined with nitric oxide to form nitrogen dioxide in an activated state. The activated NO_2 luminesces broadband visible to infrared light as it reverts to a lower energy state. Photomultiplier and associated electronics counts the photons that are proportional to the amount of NO present. Since the stream contains both NO and NO_2 , the amount of nitrogen oxide (NO_2) must first be converted to nitric oxide, NO, by passing the sample through a converter before the above ozone activation reaction is applied. The above reaction yields the amount of NO and NO_2 combined in the air sample.

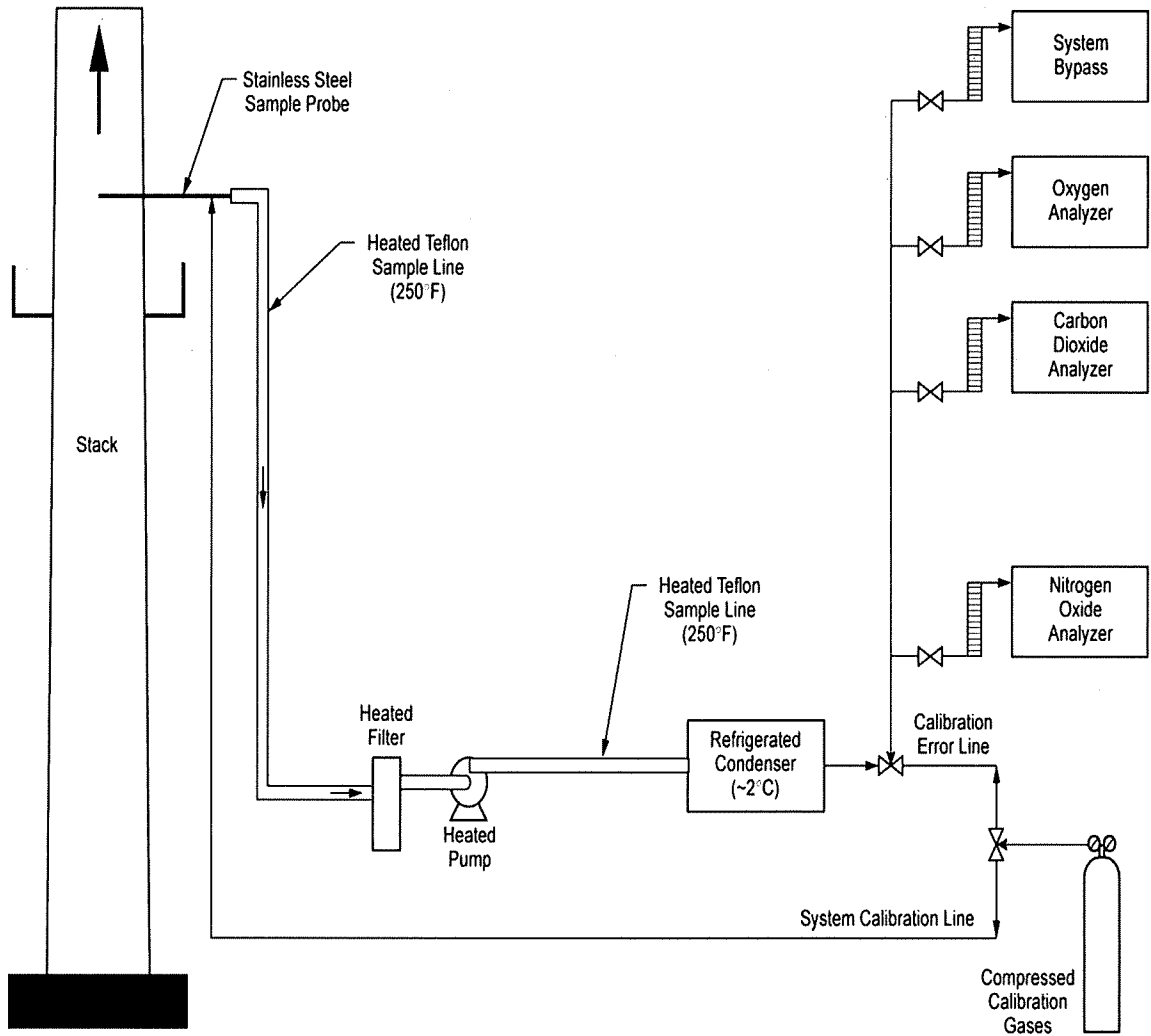
A list of analyzers utilized by AECOM for compliance testing are shown in Table 4-1.

Table 4-1. AECOM Analyzers

| AECOM Analyzers (RM) | | | | | |
|----------------------|-------|----------|-------|-----------|-------|
| Constituent | Unit | Manuf. | Model | Serial # | Span |
| Nitrogen Oxides (7E) | ppmvd | Thermo | 42c | NOx-MI902 | 0-100 |
| Oxygen (3A) | vol % | SERVOMEX | 1440 | OXC-MI90 | 0-25 |

A schematic of the instrumental sampling system is shown below in **Figure 4-2**.

Figure 4-2. Instrumental Sampling System



4.5 Visible Emissions Observations

The observer was qualified in accordance with Section 3 of Method 9 and used the following procedures for visually determining the opacity of emissions.

Position. The qualified observer shall stand at a distance sufficient to provide a clear view of the emissions with the sun oriented in the 140-degree sector to his back. Consistent with maintaining the above requirement, the observer shall, as much as possible, make his observations from a position such that his line of vision is approximately perpendicular to the plume direction and, when observing opacity of emissions from rectangular outlets (e.g., roof monitors, open baghouses, noncircular stacks), approximately perpendicular to the longer axis of the outlet. The observer's line of sight should not include more than one plume at a time when multiple stacks are involved, and in any case the observer should make his observations with his line of sight perpendicular to the longer axis of such a set of multiple stacks (e.g., stub stacks on baghouses).

Field Records. The observer shall record the name of the plant, emission location, facility type, observer's name and affiliation, and the date on a field data sheet. The estimated distance to the emission location, approximate wind direction, estimated wind speed, description of the sky condition (presence and color of clouds), and plume background are recorded on a field data sheet along with the time opacity readings are initiated and completed.

Note: The latitude and longitude on the data sheet refer to the location of the source of visible emissions.

Observations. Method 9 readings were made at the point of greatest opacity in that portion of the plume where condensed water vapor is not present. The observer did not look continuously at the plume but instead observed the plume momentarily at 15-second intervals.

Attached Steam Plumes. When condensed water vapor is present within the plume as it emerges from the emission outlet, opacity observations shall be made beyond the point in the plume at which condensed water vapor is no longer visible. The observer shall record the approximate distance from the emission outlet to the point in the plume at which the observations are made.

Detached Steam Plume. When water vapor in the plume condenses and becomes visible at a distinct distance from the emission outlet, the opacity of emissions should be evaluated at the emission outlet prior to the condensation of water vapor and the formation of the steam plume.

Recording Observations

Stack Emissions. Opacity observations for Method 9 were recorded to the nearest 5 percent at 15-second intervals on the observational record sheet. A minimum of 24 observations were recorded. The duration of this measurement must be at least 6 minutes. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period.

Data Reduction (Method 9 only). Opacity was determined as an average of 24 consecutive observations recorded at 15-second intervals. Divide the observations recorded on the record sheet into sets of 24 consecutive observations. A set is composed of any 24 consecutive observations. Sets need not be consecutive in time and in no case shall two sets overlap. For each set of 24 observations, AECOM calculated the average by summing the opacity of the 24 observations and dividing this sum by 24. If an applicable standard specifies an averaging time requiring more than 24 observations, AECOM calculated the average for all observations made during the specified time period or whatever statistical basis is specified in the permit. AECOM recorded the average opacity on the observational record sheet.

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5. Calculations

5.1 Calibration Error - Equation 7E-1

$$ACE = \frac{C_{Dir} - C_V}{CS} \times 100\%$$

C_{dir} = Measured concentration of a calibration gas (low, mid, or high) when introduced in direct calibration mode

C_V = Manufacturer certified concentration of a calibration gas (low, mid, or high)

CS = Calibration span

For oxygen, mid cal gas

| | | | |
|-----------|---|-------|---|
| C_{dir} | = | 10.10 | % |
| C_V | = | 9.93 | % |
| CS | = | 20.22 | % |

$$ACE = \frac{(10.10 - 9.93)}{20.22} \times 100\%$$

$$ACE = 0.9\%$$

For oxides of nitrogen, mid cal gas

| | | | |
|-----------|---|-------|------|
| C_{dir} | = | 30.12 | ppmv |
| C_V | = | 30.33 | ppmv |
| CS | = | 60.66 | ppmv |

$$ACE = \frac{(30.12 - 30.33)}{60.66} \times 100\%$$

$$ACE = -0.3\%$$

5.2 System Bias - Equation 7E-2

$$SB = \frac{C_S - C_{Dir}}{CS} \times 100\%$$

C_S = Measured concentration of a calibration gas (low, mid, or high) when introduced in system calibration mode

C_{Dir} = Measured concentration of a calibration gas (low, mid, or high) when introduced in direct calibration mode

CS = Calibration span

For oxygen, mid cal gas

| | | | |
|-----------|---|-------|---|
| C_S | = | 10.08 | % |
| C_{Dir} | = | 10.10 | % |
| CS | = | 20.22 | % |

$$SB = \frac{(10.08 - 10.10)}{20.22} \times 100\%$$

$$SB = -0.1\%$$

For oxides of nitrogen, mid cal gas

| | | | |
|-----------|---|-------|------|
| C_S | = | 29.92 | ppmv |
| C_{Dir} | = | 30.12 | ppmv |
| CS | = | 60.66 | ppmv |

$$SB = \frac{(29.92 - 30.12)}{60.66} \times 100\%$$

$$SB = -0.3\%$$

5.3 System Drift - Equation 7E-4

$$D = |SB_{\text{final}} - SB_i|$$

D = Drift assessment, percent of calibration span

SB_{final} = Post-run system bias, percent of calibration span

SB_i = Pre-run system bias, percent of calibration span

For oxygen, mid cal gas

| | | | |
|---------------------|---|------|---|
| SB_{Final} | = | -0.4 | % |
| Sb_i | = | -0.1 | % |

$$D = | -0.4 - (-0.1) |$$

$$D = 0.3 \%$$

For oxides of nitrogen, mid cal gas

| | | | |
|---------------------|---|------|---|
| SB_{Final} | = | -0.3 | % |
| Sb_i | = | -0.8 | % |

$$D = | -0.3 - (-0.8) |$$

$$D = 0.5 \%$$

5.4 Effluent Concentration - Equation 7E-5b

$$C_{\text{Gas}} = (C_{\text{avg}} - C_0) \frac{C_{\text{MA}}}{C_{\text{M}} - C_0}$$

C_{Gas} = Average effluent gas concentration adjusted for bias

C_{Avg} = Average unadjusted gas concentration indicated by data recorder for the test run

C_0 = Average of the initial and final system calibration bias check responses from the zero calibration gas

C_{MA} = Actual concentration of the upscale calibration gas

C_{M} = Average of initial and final system calibration bias check responses for the upscale calibration gas

For oxygen, Compliance

Run 1

| | | | |
|------------------|---|-------|---|
| C_{avg} | = | 12.44 | % |
| C_0 | = | 0.07 | % |
| C_{MA} | = | 9.93 | % |
| C_{M} | = | 10.05 | % |

$$C_{\text{gas}} = \left(12.44 - 0.07 \right) \left(\frac{9.93}{10.05 - 0.07} \right)$$

$$C_{\text{gas}} = 12.30 \quad \%$$

For oxides of nitrogen, Compliance Run 1

| | | | |
|------------------|---|-------|-----|
| C_{avg} | = | 33.18 | ppm |
| C_0 | = | 0.33 | ppm |
| C_{MA} | = | 30.33 | ppm |
| C_{M} | = | 29.77 | ppm |

$$C_{\text{gas}} = \left(33.18 - 0.33 \right) \frac{30.33}{29.77 - 0.33}$$

$$C_{\text{gas}} = 33.85 \quad \%$$

5.5 Effluent Concentration Corrected for Oxygen Concentration

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

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

P_{meas} = Measured concentration of Pollutant

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

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

For nitrogen oxides, RATA Run 1

| | | |
|----------------------|-------|-----|
| P_{meas} | 33.85 | ppm |
| $O_{2 \text{ std}}$ | 7.00 | % |
| $O_{2 \text{ meas}}$ | 12.30 | % |

$$P_{\text{Corr}} = 33.85 \times \frac{(20.90 - 7.00)}{(20.90 - 12.30)}$$

$$P_{\text{Corr}} = 54.71$$

6. Field Test Data

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Emission Summary Table
1130 CISWI Spec Mono
DuPont 1130 SPEC Mono
THROX

| Run Identification 130 CISWI Run 130 CISWI Run 130 CISWI Run | | | | | Average |
|--|-------------|-------------|-------------|--|---------|
| Flow Run Number | Flow Run 1 | Flow Run 2 | Flow Run 3 | | |
| Run Date | 6/13/23 | 6/13/23 | 6/13/23 | | |
| Run Time | 09:00-10:00 | 10:15-11:15 | 11:27-12:27 | | |
| Exhaust Gas Conditions | | | | | |
| Oxygen (% dry) | 12.30 | 12.31 | 12.28 | | 12.30 |
| Carbon Dioxide (% dry) | 6.15 | 6.17 | 6.21 | | 6.18 |
| Nitrogen Oxides | | | | | |
| Nitrogen Oxides (ppmv dry) | 33.8 | 34.4 | 34.9 | | 34.4 |
| Concentration (ppmvd @7% Oxygen) | 54.71 | 55.72 | 56.31 | | 55.5794 |