

# TEST REPORT

## COMPLIANCE EMISSION TEST HAMMERMILL NO. 5 BAGHOUSE STACK CO<sub>2</sub> SCRUBBER STACK

POET BIOREFINING  
CARO, MICHIGAN

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MAR 07 2018

AIR QUALITY DIVISION

PREPARED FOR:

**POET BIOREFINING**

1551 Empire Drive  
Caro, Michigan 48723  
Phone: 989.673.9713  
E-mail: emily.boynton@poet.com  
Attention: Ms. Emily Boynton  
Plant Manager

cc: Mr. Jacob Mackowski – jacob.mackowski@poet.com  
Mr. Charles Hauxwell – charles.hauxwell@poet.com

MAQS Project No. PROJ103928  
ARI Test Report No. 968-10  
ARI Test Plan No. 968-07 Revision 2  
ARI Proposal No. 39016  
POET Purchase Order No. CAR16695  
Test Dates: January 24 and 26, 2017



ARI Environmental, Inc. d/b/a  
Montrose Air Quality Services, LLC  
951 Old Rand Road, Unit 106  
Wauconda, Illinois 60084  
Phone: 847.487.1580 Ext. 126  
Fax: 847.487.1587  
E-mail: rburton@montrose-env.com  
Rob Burton  
Client Project Manager

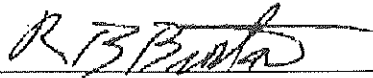
## **REPORT CERTIFICATION**

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### **STATEMENT OF CONFORMANCE AND TEST REPORT CERTIFICATION**

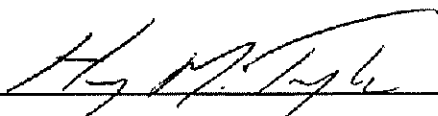
I certify, to the best of my knowledge, that this test program was conducted in a manner conforming to the criteria set forth in ASTM D7036-12: Standard Practice for Competence of Air Emission Testing Bodies, and that project management and supervision of all project related activities were performed by qualified individuals as defined by this practice.

I further certify that this test report and all attachments were prepared under my direction or supervision in accordance with the ARI Environmental, Inc. quality management system designed to ensure that qualified personnel gathered and evaluated the test information submitted. Based on my inquiry of the person or persons who performed the sampling and analysis relating to this performance test, the information submitted in this test report is, to the best of my knowledge and belief, true, accurate, and complete.



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Rob Burton, QI  
Client Project Manager  
ARI Environmental, Inc.  
d/b/a Montrose Air Quality Services, LLC



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Henry M. Taylor, QSTO  
Quality Assurance Manager  
ARI Environmental, Inc. d/b/a  
d/b/a Montrose Air Quality Services, LLC

# SECTION ONE

# Introduction and Summary

ARI Environmental, Inc. (ARI) d/b/a Montrose Air Quality Services, LLC (MAQS) was retained by POET Biorefining (POET) to conduct a compliance emission test at their facility in Caro, Michigan.

Testing was conducted to determine the concentrations and mass emission rates of particulate matter (PM), volatile organic compounds (VOC) and acetaldehyde.

Three valid 60-minute test runs were conducted on each of the following sources for the listed parameters:

Test Date	Source	Parameters	Test Methods
1/24/17	Hammermill No. 5 Baghouse Stack	PM	1, 2, 4, 17
1/26/17	CO <sub>2</sub> Scrubber Stack	VOC, Acetaldehyde	1, 2, 3A, 4, 320

The following compounds were determined using Fourier transform infrared (FTIR) instrumentation in accordance with USEPA Method 320 and ASTM Method D6348-03 to calculate the total VOC concentration:

Ethanol	Ethyl Acetate	Methanol
Acetic Acid	Formaldehyde	Acetaldehyde
Formic Acid	2-Furaldehyde	Acrolein

Where applicable, testing was conducted pursuant to the following procedures and/or regulations:

- Code of Federal Regulations, Title 40, Part 60 (40 CFR 60), Appendix A, USEPA Methods 1, 2, 3A, 4 and 17
- 40 CFR 51, Appendix M, USEPA Method 205
- 40 CFR 63, Appendix A, USEPA Method 320
- Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods
- ASTM Method D6348-03 – Determination of Gaseous Compounds by Extractive Direct Interface FTIR Spectroscopy

Mr. Jacob Mackowski of POET coordinated the test and monitored all pertinent process operations. Messrs. Rob Burton, Bryan Fotos, Brett O’Leary and Marc VanPuymbrouck of ARI performed the compliance test. Messrs. Thomas Maza and Sydney Bruestle of the Michigan Department of Environmental Quality witnessed the testing.

This report summarizes the test procedures and results of the test. Included, as appendices, is complete documentation of all calculation summaries, field data, laboratory data, ARI reference method monitoring data, FTIR data, process data, test equipment calibration data and test program qualifications.

The test results are detailed in Section 3 of this document. A summary of the average test results is presented in Table 1-1.



# SECTION ONE

# Introduction and Summary

**TABLE 1-1. SUMMARY OF AVERAGE TEST RESULTS**

Test Date	Source	Parameter	Units	Average Result	Compliance Limit
1/24/17	Hammermill No. 5 Baghouse	Total PM	gr/dscf lb/hr	0.0005 0.021	0.04
1/26/17	CO <sub>2</sub> Scrubber	Total VOC	lb/hr	<6.90	16.60
		Acetaldehyde	lb/hr	<0.02	0.80

Note: Average values labeled as "less than" identify concentrations or emission rates which include one or more compounds reported at the method detection limit.



## SECTION TWO

## Testing and Analytical Procedures

### 2.1 OVERVIEW

ARI was retained by POET to conduct a compliance emission test at their facility in Caro, Michigan. Three valid 60-minute test runs were conducted on the Hammermill No. 5 Baghouse on January 24, 2017 for PM determination. Three 60-minute test runs were conducted on the CO<sub>2</sub> Scrubber on January 26, 2017 for total VOC and acetaldehyde determination.

### 2.2 METHODOLOGY

Where applicable, test procedures and sampling methodology followed 40 CFR 60, Appendix A, USEPA Methods 1, 2, 3A, 4 and 17; 40 CFR 51, Appendix M, USEPA Method 205; 40 CFR 63, Appendix A, USEPA Method 320; ASTM Method D6348-03; and the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods.

#### 2.2.1 Sampling Locations (USEPA Method 1)

The velocity sampling points were determined following USEPA Method 1. The sampling locations and number of velocity sampling points were as follows:

Stack Sampling Location	Stack Diameter (inches)	Port Location Upstream from Disturbance (inches)	Port Location Downstream from Disturbance (inches)	No. of Ports	Sampling Points per Port	Total Points
Hammermill No. 5 Baghouse	21.25	300	67	2	12	24
CO <sub>2</sub> Scrubber	23.25	15	74	2	8	16

Cyclonic flow checks were conducted to demonstrate that cyclonic flow conditions did not exist at the sampling locations.

#### 2.2.2 Gas Velocity and Volumetric Flow Rate Determination (USEPA Method 2)

Gas velocity and volumetric flow rate were determined following USEPA Method 2 procedures. Velocity traverses were performed using a Type-S pitot tube with the velocity head pressure measured on a Dwyer oil gauge inclined manometer to the nearest 0.01-in. H<sub>2</sub>O. Temperature measurements were performed with a chromel-alumel thermocouple connected to a digital direct read-out potentiometer.

The Hammermill No. 5 Baghouse stack gas composition is equivalent to ambient air; therefore, a dry molecular weight of 29.0 lb/lb-mole was used for the gas density and flow calculations as stated in USEPA Method 2.

#### 2.2.3 Molecular Weight (USEPA Method 3A)

At the CO<sub>2</sub> Scrubber stack, the carbon dioxide (CO<sub>2</sub>) and oxygen (O<sub>2</sub>) concentrations were determined in accordance with USEPA Method 3A procedures using ARI's Servomex Model 1440 combination paramagnetic O<sub>2</sub> and non-dispersive infrared CO<sub>2</sub> analyzer.

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As shown in Figure 2-1, ARI's Method 3A sampling system consisted of a heated probe followed by a calibration tee. The probe system was connected to a heated Teflon sampling line that transported the gas sample to an electronic chiller to remove moisture. The dry sample gas was then transported to a manifold system by a Teflon-lined sample pump and Teflon sample line. The manifold was connected with sample gas intake lines for ARI's analyzers.

The sampling system was calibrated with applicable zero, mid-range and high-range gases as specified in USEPA Method 3A. The calibration gases were generated from Protocol 1 calibration gases using an EnviroNics Model 4040 Gas Dilution System. The gas dilution system was verified on site in accordance with USEPA Method 205.

Response time, calibration error and measurement system bias tests were performed prior to testing, and a pre/post calibration drift test was performed on each analyzer. The average zero and calibration drift values obtained during each test run were used to correct the analyzer's raw data for instrument zero and drift for each respective test run.

The data was collected at 15-second intervals by ARI's data acquisition system that consisted of a datalogger connected to a computer for digital data storage and reduction.

### **2.2.4 Moisture Content (USEPA Method 4)**

The stack gas moisture content at the baghouse was determined in accordance with USEPA Method 4 and combined with the PM sampling train.

At the CO<sub>2</sub> Scrubber, FTIR measurements were used for determination of stack gas moisture content per USEPA Method 4, Section 16.3.

### **2.2.5 PM Determination (USEPA Method 17)**

PM was determined following the procedures described in USEPA Method 17 - Determination of Particulate Emissions from Stationary Sources (In-Stack Filtration Method).

#### **2.2.5.1 Sampling Apparatus**

Assembled by ARI, the sampling train consisted of the following:

Nozzle - Stainless steel (316) with sharp, tapered leading edge.

Probe - Stainless steel liner and outer sheath.

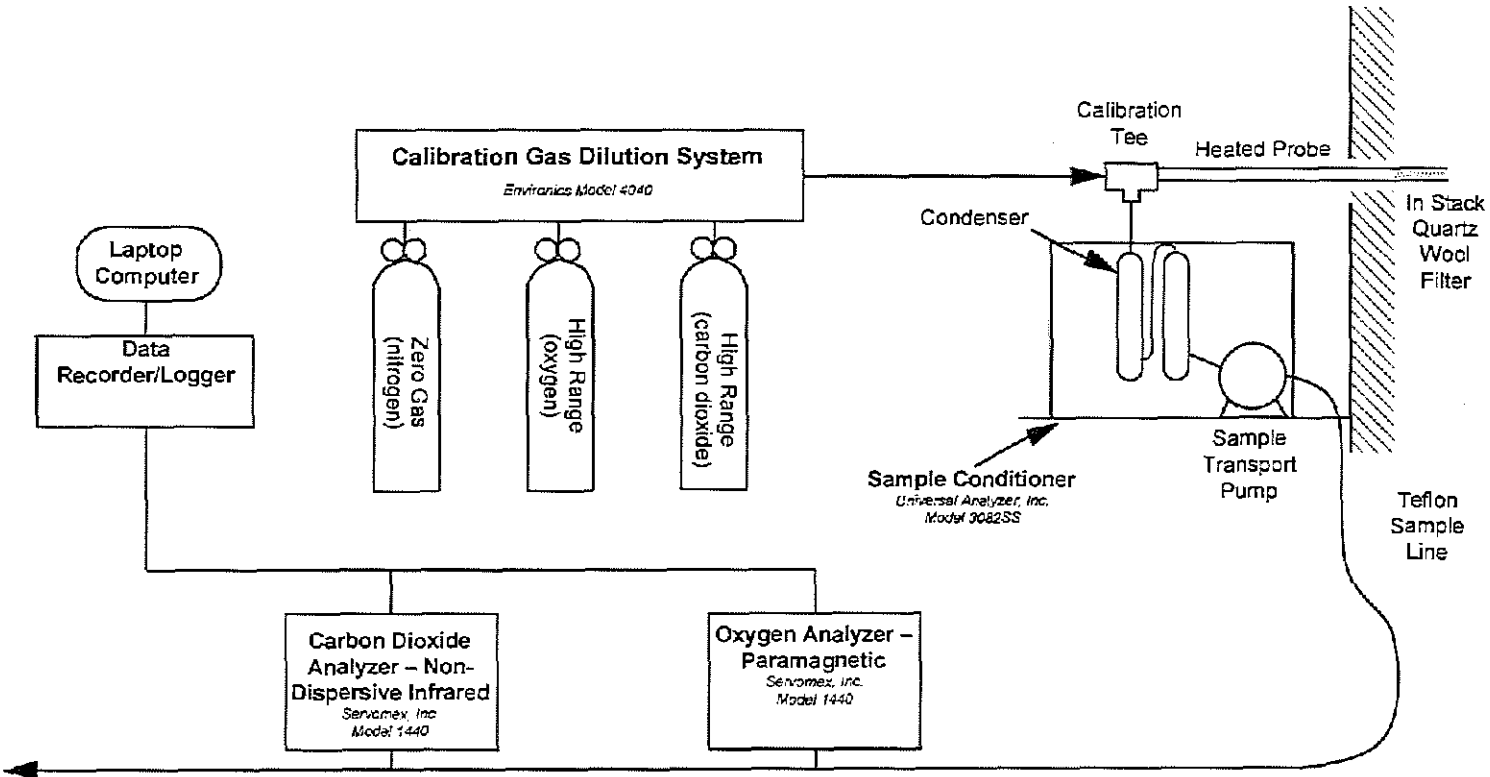
Pitot Tube - Type-S, attached to probe for monitoring stack gas velocity.

In-Stack Filter Holder - Method 17 filter housing containing 47-mm glass-fiber filter.

Draft Gauge - Inclined manometer with a readability of 0.01-in. H<sub>2</sub>O in the 0 to 1-in. range.

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**FIGURE 2-1. ARI REFERENCE METHOD O<sub>2</sub> AND CO<sub>2</sub> SAMPLING SYSTEM**

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## Testing and Analytical Procedures

Impingers - Four impingers connected in series with glass ball joints. The second impinger was of the Greenburg-Smith design with a standard tip. The first, third and fourth impingers were of the Greenburg-Smith design, but modified by replacing the standard tip with a ½-in. i.d. glass tube extending to within ½-in. of the bottom of the impinger flask.

Metering System - Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5°F, dry gas meter with ±2 percent accuracy and related equipment as required to maintain an isokinetic sampling rate and to determine sample volume.

Barometer - Mercury barometer capable of measuring atmospheric pressure to within ±0.1-in. Hg.

### 2.2.5.2 Sampling Procedures

After the sampling site and minimum number of traverse points were selected, the stack pressure, temperature, moisture and range of velocity head were measured according to procedures described in USEPA Methods 1 through 4. The sampling train was set up with the probe, in-stack filter holder and impingers as shown in Figure 2-2. The first and second impingers each contained 100 milliliters (mL) of deionized/distilled water, the third impinger was empty and the fourth impinger contained 200 grams of silica gel. The impingers were placed in an insulated sampling bucket and ice water bath to reduce the sample gas temperature to ≤68°F upon exiting the last impinger during sampling.

The sampling train was leak-checked at the sampling site by plugging the inlet to the nozzle and pulling a vacuum of 15-in. Hg. A leakage rate of less than 0.02 ft<sup>3</sup>/min, at a vacuum of 15-in. Hg, is considered acceptable. At the completion of each test run, the sampling train was again leak-checked by the same procedure, but at the highest vacuum attained during the test run. Both pre-test and post-test leak checks of the pitot tube and lines were made for each test run. These checks were made by blowing into the impact opening of the pitot tube until 3 or more inches of water column was achieved on the manometer and then capping the impact opening. The leak check is considered successful if the manometer level holds steady for more than 15 seconds. The static pressure side of the pitot tube was leak-checked by the same procedure, except suction was used to obtain the 3-in. H<sub>2</sub>O manometer reading.

During sampling, stack gas and sampling train data were recorded at specified intervals. Isokinetic sampling rates were set throughout the sampling period with the aid of a programmable calculator.

### 2.2.5.3 Sample Recovery Procedures

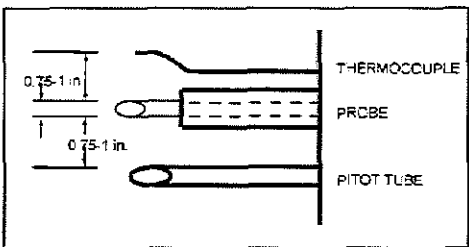
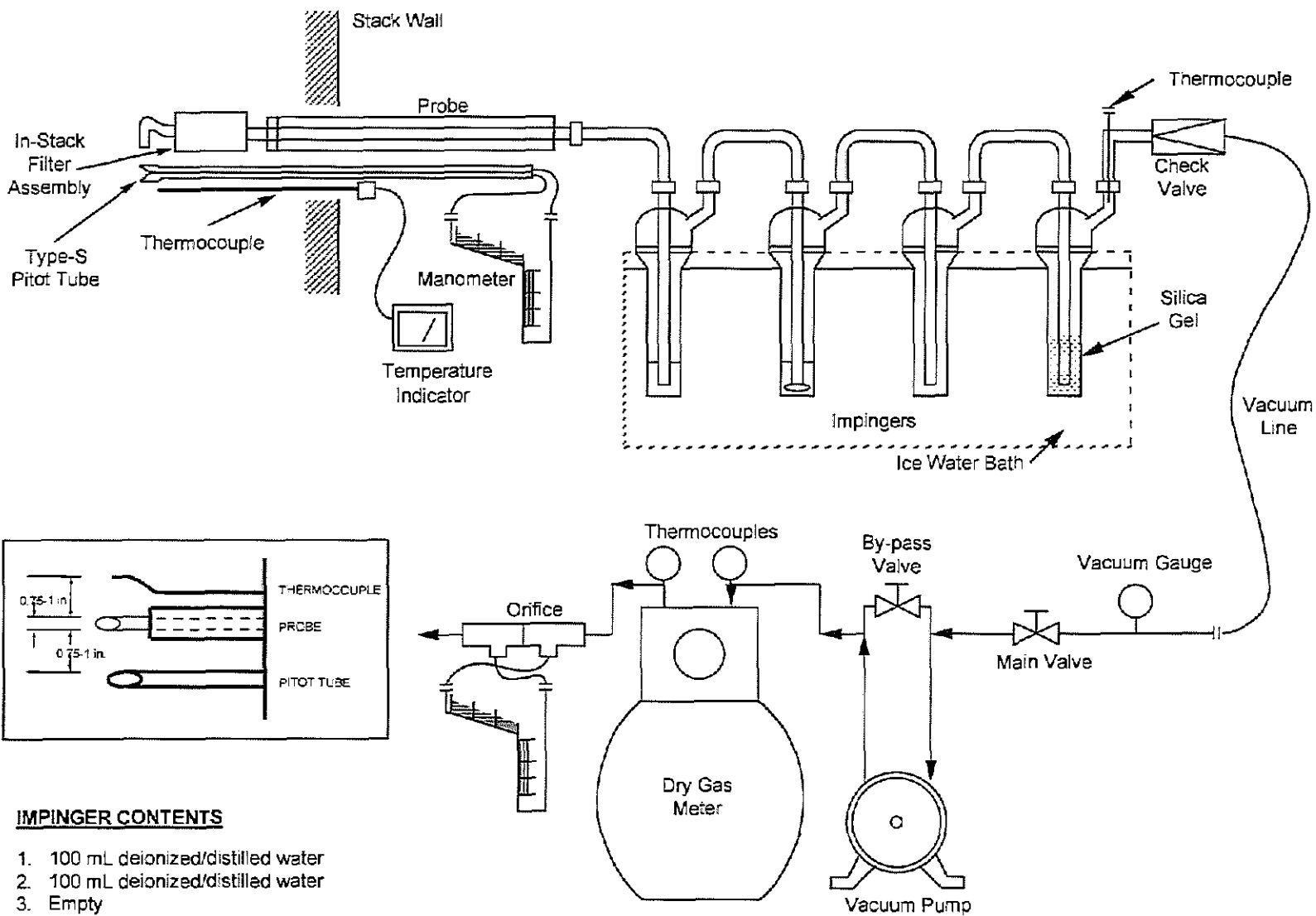
After sampling was completed and the final leak checks were performed, the filter holder and probe (front-half) were disconnected from the impinger train. The sample fractions were recovered as follows:

Container 1 - The filter was removed from its holder, placed in a labeled dish and sealed.



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**IMPINGER CONTENTS**

1. 100 mL deionized/distilled water
2. 100 mL deionized/distilled water
3. Empty
4. 200 g silica gel

**FIGURE 2-2. USEPA METHOD 17 PARTICULATE MATTER SAMPLING TRAIN**



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Container 2 - PM was removed from the nozzle and filter holder exposed surfaces prior to the filter with the aid of a brush and acetone rinsing. The washings were placed in a glass bottle, sealed and labeled. The liquid level was marked after the container was sealed.

Container 3 - A minimum of 200 mL of acetone was taken for the blank analysis. The blank was obtained and treated in a similar manner as the contents of Container 2.

The contents of the first, second and third impingers were measured for volume and discarded. The contents of the fourth impinger (silica gel) were placed in a polyethylene bottle for subsequent weighing to the nearest 0.1 gram.

### 2.2.5.4 Analytical Procedures

The analytical procedures were as follows:

Container 1 - The filter was placed in a desiccator for 24 hours and weighed to a constant weight to the nearest 0.1 mg.

Container 2 - The nozzle and upstream portion of the filter holder washings were transferred to a tared beaker and dried at ambient temperature. Then the beaker was placed in a desiccator for 24 hours and weighed to a constant weight to the nearest 0.1 mg.

Container 3 - The acetone blank was transferred to a tared beaker and dried at ambient temperature. Then the beaker was placed in a desiccator for 24 hours and weighed to a constant weight to the nearest 0.1 mg.

The term "constant weight" means a difference of no more than 0.5 mg or 1 percent of the total weight less tare weight, whichever is greater between two consecutive readings, with no less than 6 hours of desiccation between weighings.

### 2.2.6 Gas Dilution System Verification (USEPA Method 205)

All diluted calibration standards were prepared using an EnviroNics Model 4040 Gas Dilution System that was verified by a field evaluation at the job site following the requirements of USEPA Method 205.

ARI's Servomex Model 1440 paramagnetic O<sub>2</sub> gas analyzer was calibrated following USEPA Method 3A procedures using O<sub>2</sub> in nitrogen standards. After the calibration procedure was complete, two diluted standards and an EPA Protocol 1 standard were alternately introduced in triplicate, and an average instrument response was calculated for each standard. No single response differed by more than  $\pm 2\%$  from the average response for each standard. The difference between the instrument average and the predicted concentration was less than  $\pm 2\%$  for each diluted standard. The difference between the certified gas concentration and the average instrument response for the EPA Protocol 1 standard was less than  $\pm 2\%$ .

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### ***2.2.7 FTIR Single Instrument Spiking Validation (USEPA Method 301)***

ARI has performed several USEPA Method 301 spiking studies to validate the use of FTIR techniques to accurately measure the concentrations of acetaldehyde, acrolein, formaldehyde and methanol from scrubber stacks.

The results of the validation studies were used to determine if the FTIR procedures are valid for this type of stationary source. USEPA Method 320 allows the validation of FTIR-based measurements by a pair-wise comparison between the results of a single FTIR system.

The results of the validation studies indicated an accuracy of  $<\pm 2\%$  for the specific compounds; therefore, a bias correction factor is not needed. All of the statistical criteria in the method were met. The validation studies are on file at ARI.

### ***2.2.8 VOC and Acetaldehyde Determination using FTIR Spectroscopy (USEPA Method 320)***

VOC and acetaldehyde sampling was conducted using FTIR instrumentation following USEPA Method 320 and ASTM Method D6348-03.

ARI used an MKS Model MultiGas 2030 FTIR to measure the pollutant concentrations. The MultiGas 2030 analyzer was composed of a 2102 process FTIR spectrometer, a high optical throughput sampling cell, analysis software and a quantitative spectral library. The analyzer collects high resolution spectra in the mid infrared spectral region (400 to 4,000  $\text{cm}^{-1}$ ), which are analyzed using the quantitative spectral library. This provides an accurate, highly sensitive measurement of gases and vapors.

As shown in Figure 2-3, the sample delivery system consisted of a stainless steel sampling probe, calibration tee, Teflon sampling line, fast loop bypass pump, dilution system and sample manifold. The gas sample was continuously extracted from the source at approximately 8 liters per minute.

It should be noted that the main principles and calibration procedures of USEPA Method 320 were followed. USEPA Method 320 specifies a number of analytical uncertainty parameters that the analyst calculated to characterize the FTIR system performance. However, this did not provide analytical detection limits. To calculate the method detection limit (MDL) for the target compounds, the guidelines in Appendix B of 40 CFR 136 were followed. With this, the Student t-test is used to calculate the MDL for each analyte at a 99% confidence level. This follows USEPA guidelines for reporting of zeroes or non-detects and also meets the NELAC requirements for determination of MDL values.

The MKS software calculates the analytical error of the FTIR measurement which includes the root mean standard deviation (RMSD). The concentration uncertainty reported by MKS is called the standard error of estimated concentration (SEC) and is also known as the marginal standard deviation. The uncertainties in the concentration are proportional to the square root of the sums of the squares of the residual. After the residual spectrum is obtained, which is called R, the error variance for the case of a single reference spectrum is calculated by the software.

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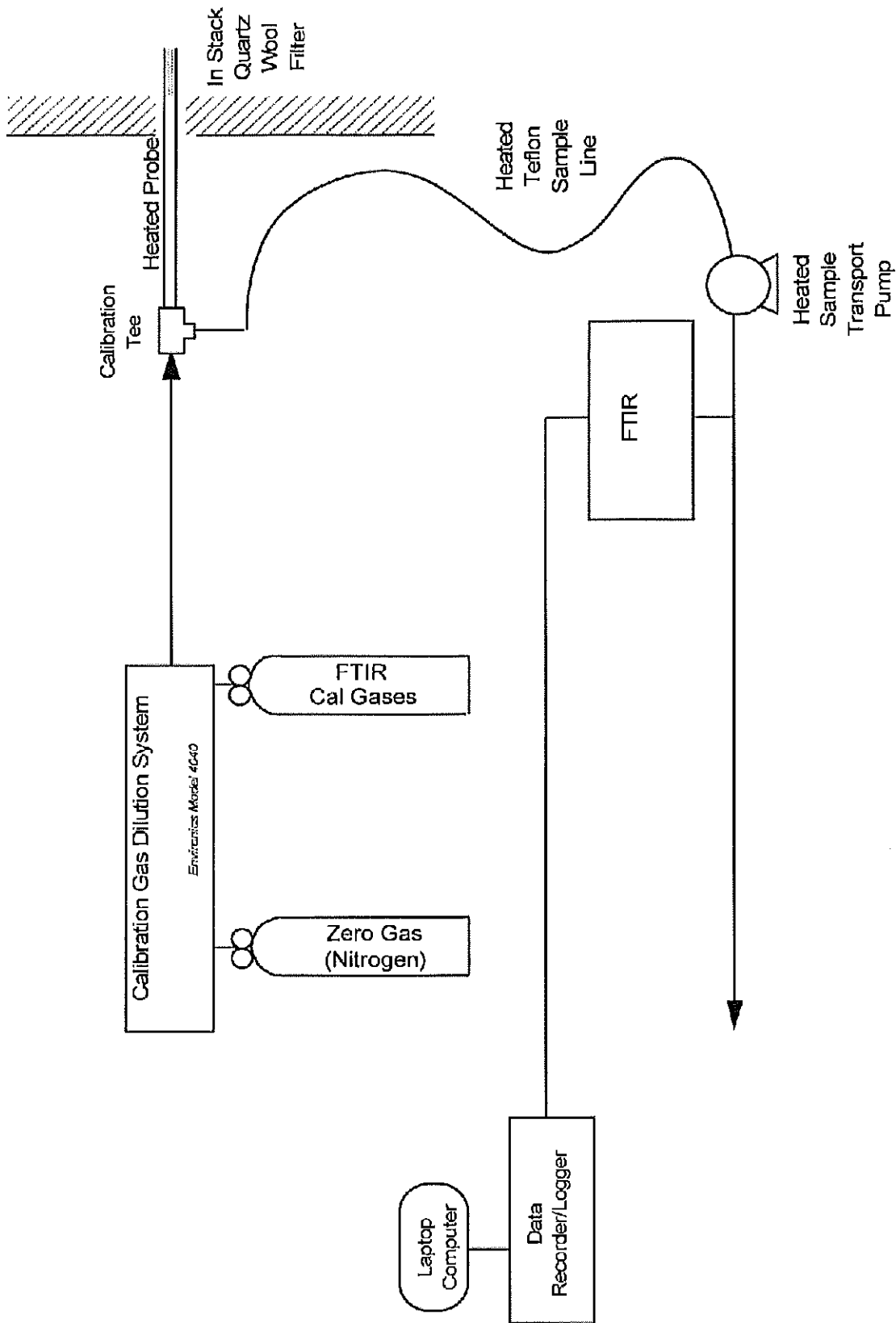


FIGURE 2-3. FTIR SAMPLING SYSTEM

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## **Testing and Analytical Procedures**

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Independent calculations of optical path length were not performed because the instrument has a fixed path of 5.11 meters. A signal to noise ratio test (S/N) was performed using MKS software to verify instrument performance.

Performance parameters measured included signal to noise tests, noise equivalent absorbance (NEA), detector linearity, background spectra, potential interferences and cell and system leakage.

Quality assurance procedures included baseline measurement with ultra-high purity nitrogen, measurement of a calibration transfer standard (~100 ppm ethylene), direct analyte calibration measurements and measurements to determine baseline shift. SF<sub>6</sub> was also used as a tracer gas in the calibration gases to evaluate dilution ratios and verify the sample delivery system integrity. A dynamic matrix spike was performed using acetaldehyde and SF<sub>6</sub> as a tracer gas.

The general FTIR field sampling procedure was as follows:

### **PRE-TEST**

- 1) Background spectrum
  - Evaluate diagnostics of the instrumentation
- 2) Baseline (cylinder UHP-N<sub>2</sub> for zero check)
  - Determine the level of background noise
  - Observe spectrum for baseline tilt, i.e., indicates vibrations/perturbations affecting instrument
- 3) Calibration Transfer Standard (cylinder 100-ppm ethylene for span check)
  - Determine level of response to evaluate the spectral response and stability of the instrument
  - Create a field Reference Spectrum
- 4) Baseline Evaluation
  - Note baseline flush/clean out FTIR sample cell
  - Observe spectrum for baseline tilt
- 5) Collection of spectra stack gas
  - Determine stack gas analyte concentrations
- 6) Measurement of analyte calibration gases
- 7) Perform Dynamic Matrix Spiking (recovery must be 70 to 130%)

### **TEST (REPEAT EACH RUN)**

- 1) Baseline Determination
- 2) Measurement of Calibration Transfer Standard
- 3) Collect sequential spectra of stack gas
- 4) Baseline Determination
- 5) Measurement of Calibration Transfer Standard

### **POST-TEST**

- 1) Baseline Determination
- 2) Measurement of Calibration Transfer Standard (i.e. span check)
- 3) Measurement of analyte calibration gas (optional)



## **SECTION THREE**

## **Test Results**

The compliance emission test results are presented in Tables 3-1 and 3-2.

The calculation summaries, field data, laboratory data, ARI reference method monitoring data, FTIR data, process data, calibration data and test program qualifications are included in the appendices.

### ***DISCUSSION***

The first test run conducted on the Hammermill No. 5 Baghouse was excluded due to a sampling probe stack temperature thermocouple malfunction. An additional test run was conducted, and the results of Test Run Nos. 2, 3 and 4 are presented in Table 3-1. The field and laboratory data for the first test run are included in the appendices.

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# SECTION THREE

# Test Results

**TABLE 3-1. HAMMERMILL NO. 5 BAGHOUSE PM TEST RESULTS**

TEST RUN NO. :	2	3	4	Average	Compliance Limit
TEST DATE :	1/24/2017	1/24/2017	1/24/2017		
TEST TIME :	11:20-12:21	12:33-13:35	13:55-14:57		
<b><u>Stack Gas Parameters</u></b>					
Temperature, °F	76.7	74.3	75.3	75.4	
Velocity, av. ft/sec	35.1	35.0	34.9	35.0	
Volumetric flow, acfm	5,191	5,174	5,155	5,173	
Volumetric flow, scfm	4,945	4,950	4,923	4,939	
Volumetric flow, dscfm	4,893	4,903	4,865	4,887	
Volumetric flow, dscfh	293,576	294,195	291,911	293,227	
Moisture, av. % vol	1.0	0.9	1.2	1.1	
Carbon dioxide, av. % vol	0.0	0.0	0.0	0.0	
Oxygen, av. % vol	20.9	20.9	20.9	20.9	
<b><u>Particulate Sample</u></b>					
Time, min.	60.0	60.0	60.0	60.0	
Volume, dscf	38.812	41.078	38.396	39.429	
Filterable PM, mg	1.1	2.0	0.9	1.3	
Isokinetic ratio, %	99.5	105.0	99.0	101.2	
<b><u>Total Particulate Matter</u></b>					
Concentration					
grains/dscf	0.0004	0.0007	0.0003	0.0005	
x 10 <sup>-6</sup> lb/dscf	0.062	0.105	0.049	0.072	
Emission rate					
lb/hr	0.018	0.031	0.014	0.021	0.04



# SECTION THREE

# Test Results

**TABLE 3-2. CO<sub>2</sub> SCRUBBER VOC AND ACETALDEHYDE TEST RESULTS**

TEST RUN NO. :	1	2	3		
TEST DATE :	1/26/2017	1/26/2017	1/26/2017		Compliance
TEST TIME :	<u>08:10-09:10</u>	<u>09:40-10:40</u>	<u>11:07-12:07</u>	<u>Average</u>	<u>Limit</u>
<b><u>Stack Gas Parameters</u></b>					
Temperature, °F	57.9	58.5	59.9	58.8	
Velocity, av. ft/sec	43.6	42.1	45.2	43.6	
Volumetric flow, acfm	7,718	7,451	7,988	7,719	
Volumetric flow, scfm	7,535	7,266	7,767	7,523	
Volumetric flow, dscfh	444,905	428,902	458,215	444,007	
Moisture, av. % vol	1.6	1.6	1.7	1.6	
Carbon dioxide, av. % vol	93.1	93.2	93.5	93.3	
Oxygen, av. % vol	1.4	1.3	1.3	1.4	
<b><u>Total VOC (FTIR)</u></b>					
Emission rate, lb/hr	<6.65	<6.52	<7.53	<6.90	16.60
<b><u>HAP - Acetaldehyde</u></b>					
Concentration					
ppmv wb	<0.45	<0.45	<0.45	<0.45	
x10 <sup>-6</sup> lb/scf	<0.052	<0.052	<0.052	<0.052	
Emission rate					
lb/hr	<0.02	<0.02	<0.02	<0.02	0.80