

**PASSIVE FOURIER TRANSFORM INFRARED TECHNOLOGY (FTIR)  
EVALUATION OF P001 PROCESS CONTROL DEVICE  
AT THE  
INEOS ABS (USA) CORPORATION  
ADDYSTON, OHIO FACILITY**

**July 2010**

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## 1.0 SUMMARY OF EVALUATION PROGRAM

Pursuant to the Consent Decree (Civil Action No. 1:09-CV-545) between U.S. Environmental Protection Agency (EPA) vs. INEOS ABS (USA) Corporation (INEOS ABS) and LANXESS Corporation, INEOS ABS performed a Passive FTIR evaluation on the P001 Process control device at the INEOS ABS Addyston, Ohio facility. The purpose of the evaluation was to determine the appropriate Net Heating Value of Flare Gas (NHVFG) limit to be used to operate the control device to ensure 99% control efficiency.

The Passive FTIR (PFTIR) evaluation was performed on the P001 process air pollution control device (the Flare). The PFTIR method was used to estimate emissions from the Flare by measuring carbon dioxide (CO<sub>2</sub>), carbon monoxide (CO) and total hydrocarbons (THC) in the Flare output stream (the area above the flame of the Flare) at varying operating conditions and NHVFG values. The PFTIR evaluation was a comparative test only as it is still a developing technology for measuring flare emissions.

The EPA has proposed that a NHVFG of 200 British Thermal Units per standard cubic feet (BTU/scf) yields a control efficiency of 99%. This PFTIR evaluation compared environmental performance at different NHVFG values so as to determine the range of NHVFG that provides optimum environmental performance for operation of the Addyston Flare.

INEOS ABS contracted with Industrial Monitor and Control Corporation (IMACC) to perform the PFTIR evaluation of the Flare. The test was performed in a timely manner on November 3 through November 5, 2009. The following personnel from EPA and Hamilton County Department of Environmental Services (HCDES) observed the evaluation:

- Cary Secrest, EPA Headquarters (Washington, D.C.)
- Brian Dickens, EPA Region V (Chicago)
- Mike Kramer, HCDES

The evaluation pursuant to the Consent Decree was only being used to provide information to the EPA Office of Enforcement and Compliance Assurance (OECA), Director of Air Enforcement of the Office of Civil Enforcement (OCE) to aid in his/her decision in the establishment of a newly defined lower NHVFG limit to be used to control the P001 Process Flare. This evaluation was not used as a means to determine compliance with the facility's Title V Permit.

## **2.0 SOURCE DESCRIPTION**

### **2.1 PROCESS DESCRIPTION**

Figure 2-1 illustrates the basic processing steps in the P001 Process. The general steps in the process include:

- Reacting 1,3-butadiene with other ingredients in kettles to produce a rubber latex;
- Cooling the intermediate latex; and
- Holding the intermediate latex in preparation for further processing.

The P001 process is a batch process. Process emissions from the P001 process are routed to the Flare or the boilers for destruction. Process emissions are generated as the materials react, while materials are cooling in process tanks, or when materials are breathing while in storage tanks. Process emissions sent to the Flare are routed through the seal tank on the way to the Flare (see Figure 2-2). Wastewaters from the process are routed to the chemical sewer for further treatment at the facility's wastewater treatment plant.

### **2.2 CONTROL EQUIPMENT DESCRIPTION**

The Flare is the control device that was evaluated during this test. The Flare's steam-assisted tip was manufactured by the John Zink Company, LLC (Zink) (Model # EEF QS-16) and is equipped with a Zink airrestor purge reduction device (Model # EEF-AR-16). Figure 2-2 shows the P001 process vent overview and Figure 2-3 shows the Flare layout.

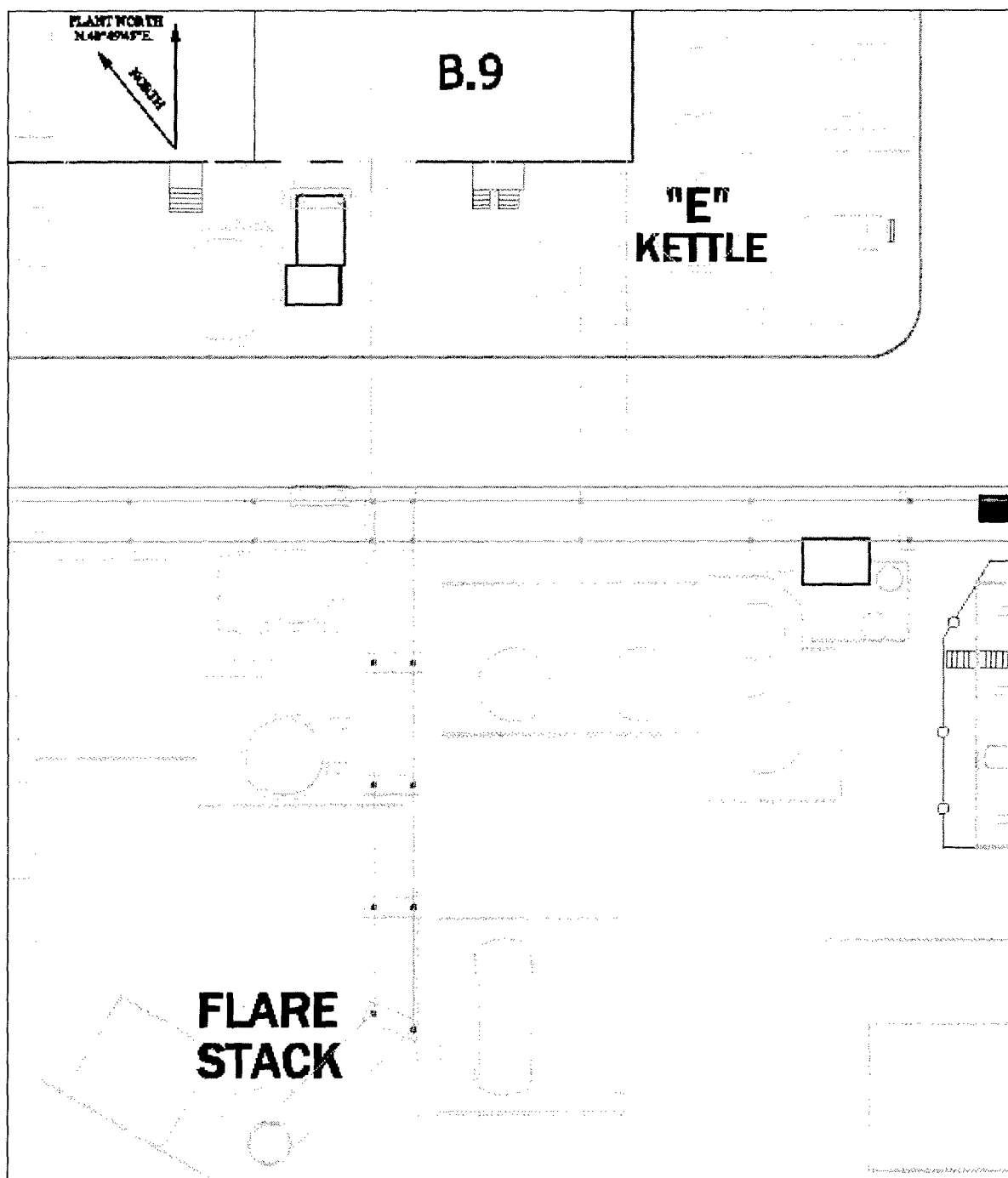
Several monitoring instruments are used to calculate parameters that have been identified as influencing flare efficiency. The amount of steam flow to the Flare tip and the amount of natural gas added to the Vent Gas are direct measurements from flow meters in the field. A GE Sensing DigitalFlow Panametrics Ultrasonic Flowmeter (Panametrics Model # GF868) is located in the Flare system to measure velocity and molecular weight of the stream from the seal tank to the Flare. These measured values are used to calculate the amount of 1,3-butadiene in the stream, total Vent Gas flow rate and subsequent heat value of the Vent Gas going to the Flare. In addition, these values can be used to calculate the steam-to-Vent Gas ratio and the lower NHVFG.

## 2.3 SAMPLING LOCATION

The PFTIR evaluation was performed on the P001 Process Flare. The location of the FTIR equipment was on an elevated platform 165 feet to the east of the Flare (labeled “E” KETTLE on Figure 2-4). This location gave a direct line of sight to the Flare tip and plume and helped to minimize interferences from surrounding buildings and process equipment. However, one limitation this created was experienced whenever the wind direction was blowing the plume towards or away from the FTIR camera. This made it more difficult to keep the camera focused on the best part of the plume to give a good indication of composition.

The PFTIR measurements were intended to be taken in the centerline of the plume with the plume filling the entire field of view of the FTIR equipment. A thermal imaging camera was used to aid the technicians in making constant manual adjustments of the position of the FTIR camera by attempting to locate the hottest area above the flame in the Flare plume that would allow for a full field of vision for the FTIR equipment. The zone monitored was intended to be above the combustion zone so the majority of combustion was complete, yet low enough so the temperature was above 200°C to allow detection (within two meters of the flame).

Figure 2-4 Sampling Location



### **3.0 EVALUATION PROGRAM SUMMARY AND RESULTS**

#### **3.1 OBJECTIVES AND TEST MATRIX**

The purpose of the PFTIR evaluation was to determine the appropriate lower NHVFG limit to be used to operate the P001 Process Flare. The PFTIR method was used to estimate the emissions from the Flare by measuring CO<sub>2</sub>, CO and THC in the Flare output stream (the area above the flame of the Flare) at varying operating conditions and NHVFG values.

During the evaluation, operating scenarios of the Flare were varied in order to determine which lower NHVFG limit is the most appropriate, i.e. most efficient. Operating scenarios involved varying the flow rates and composition of the steam and the Vent Gas stream. The purpose of this evaluation program was to run scenarios to bracket conditions and to compare emissions at different scenarios. The ultimate goal was to compare the emissions at different scenarios and recommend the future lower limit of NHVFG. It is proposed that any scenarios with lower emissions than those seen at the 200 BTU/scf scenarios would be considered to be representative of control efficiency higher than 99% as the EPA has stated that a 200 BTU/scf NHVFG would achieve a 99% efficiency.

Table 3-1 presents the sampling and analytical evaluation matrix that was proposed to be used to help establish the appropriate lower NHVFG limit. The runs were divided into four different flow rates of 1,3-butadiene. Different NHVFG were proposed within each of these flow rates.

#### **3.2 FIELD TEST CHANGES AND PROBLEMS**

At the beginning of the evaluation, it became apparent that achieving and keeping steady flows to the Flare would be challenging. Due to these complications, several runs were modified and not performed in the order that was proposed. These runs were modified after consulting and receiving the approval of the EPA and HCDES who were observing the evaluation on site. The modified sampling and analytical evaluation matrix that was followed during the field test is shown in Table 3-2.

In particular, during some test runs, 1,3-butadiene flows were modified (lower or higher than proposed). As a consequence, the exit velocities were also either lower or higher than proposed. However, the target NHVFG for the test runs was not impacted as this was the value that was the controlling parameter to start each test run. The flows of 1,3-butadiene, natural gas, and steam were modified for each test run to achieve the target NHVFG. Once the target NHVFG was achieved the test was begun. In addition, two test runs (Runs 1B and 12) were shortened to less than twenty minutes of sampling time due to significant changes in 1,3-butadiene flow rates. Runs 6, 7, 9 and 17A were shortened under agreement with EPA and HCDES as good data was determined to have been collected in less than 20 minutes.

IMACC performed daily calibration checks using the black body source. IMACC is working on technology to do calibration tests for the primary gas of concern (1,3-butadiene in the case of these tests) by using a flow tube with that gas in it. This approach was not used for these tests as the technology was not available for 1,3-butadiene at the time of the evaluation.

Initial instrument set-up was performed on November 3, 2010 and shots of the sky were made during calibration. Problems with the sensor becoming saturated were experienced when the sensor was exposed to too much sunlight and a different sensor tube had to be installed.

On the night of November 3, 2010, the instrument got too cold and difficulties were experienced with sluggish movement of sensor elements which influenced analyzer capability the next day. These problems were minimized by providing a heat source for the sensor housing for subsequent runs.

### 3.3 SUMMARY OF RESULTS

Upon review of the PFTIR test data, it was discovered that there was a problem with some of the data generated by the Panametrics monitoring system. After a great deal of investigation and extensive discussions with the manufacturer of the Panametrics monitoring system, it was discovered that a fixed (80%) nitrogen concentration value differed greatly from actual concentrations and hence impacted the resulting molecular weight calculation performed by the Panametrics.

Upon this discovery, INEOS ABS acted promptly and diligently to determine the nature of the problem and to seek a remedy. In this regard, it was discovered that the manufacturer of the monitoring system (GE Sensing) had developed an algorithm to correct this problem, but the manufacturer initially refused to provide this proprietary solution. Finally, in exchange for a fee and a confidentiality agreement, INEOS ABS obtained the algorithm. The algorithm is extremely complex and has taken a significant period of time to make the necessary corrections to the data. INEOS ABS is in the process of converting the test data in order to evaluate the appropriate NHVFG limit to be used to operate the P001 Process Flare.

Please see the timeline of events below. As such, this report will be amended to include the results of the evaluation by August 6, 2010.

<u>Date</u>	<u>Event</u>
3/22/10	Conference call between EPA, HCDES, and INEOS ABS regarding correct method to calculate 1,3-butadiene flow.
4/15/10	Conference call between EPA, HCDES, and INEOS ABS continuing discussion on correct method to calculate 1,3-butadiene flow and discussion regarding the Panametrics with regards to nitrogen concentrations and molecular weight.
Week of 04/12/10	Began discussion with GE Sensing to obtain algorithm to correct molecular weight.
4/21/10	Conference call between EPA, HCDES, and INEOS ABS continuing discussion on correct method to calculate 1,3-butadiene flow and the Panametrics with regards to nitrogen concentrations and molecular weight.
4/22/10	GE Sensing agreed that it may be possible to supply an algorithm to correct molecular weight.
5/3/10	Conference call between EPA, HCDES, and INEOS ABS continuing discussion on



correct method to calculate 1,3-butadiene flow and the Panametrics with regards to nitrogen concentrations and molecular weight. Agreed that % mass verses % mol would be used to calculate 1,3-butadiene flow.

5/6/10 GE Sensing agreed to supply INEOS ABS with the algorithm for a fee and a Confidentiality agreement. GE Sensing to supply proposal to INEOS ABS.

5/10/10 Conference call between EPA, HCDES, and INEOS ABS continuing discussion on the Panametrics with regards to nitrogen concentrations and molecular weight.

5/13/10 INEOS ABS received proposal from GE Sensing.

5/14/10 INEOS ABS submitted Purchase Order to GE Sensing.

5/25/10 INEOS ABS received algorithm from GE Sensing.

**Table 3-3 Data Summary Table  
INEOS ABS – Addyston, Ohio**

Test Run	Proposed Exit Velocity (ft/min)	Actual Exit Velocity During Test Run (ft/min)	Proposed Butadiene Flow (lb/hr)	Actual Butadiene Flow During Test Run (lb/hr)	Proposed NHVFG (BTU/scf)	Actual NHVFG (BTU/scf)	PFTIR Estimated Efficiency (%)
1	110	<i>TO</i>	300	<i>TO</i>	120	<i>TO</i>	<i>TO</i>
1A	NA	<i>BE</i>	NA	<i>BE</i>	NA	<i>BE</i>	<i>BE</i>
1B	NA	<i>PROVIDED</i>	NA	<i>PROVIDED</i>	NA	<i>PROVIDED</i>	<i>PROVIDED</i>
2	120	<i>AFTER</i>	300	<i>AFTER</i>	200	<i>AFTER</i>	<i>AFTER</i>
3	110	<i>THE</i>	300	<i>THE</i>	150	<i>THE</i>	<i>THE</i>
4	160	<i>TEST</i>	300	<i>TEST</i>	180	<i>TEST</i>	<i>TEST</i>
5	110	<i>DATA</i>	300	<i>DATA</i>	230	<i>DATA</i>	<i>DATA</i>
6	180	<i>IS</i>	600	<i>IS</i>	150	<i>IS</i>	<i>IS</i>
7	270	<i>CONVERTED</i>	600	<i>CONVERTED</i>	210	<i>CONVERTED</i>	<i>CONVERTED</i>
8	250		600		190		
9	250		600		230		
10	75		50		120		
11	90		50		200		
12	40		50		200		
13	90		50		140		
14	80		50		160		
15	80		50		180		
16	90		50		220		
17	50		10		140		
17A	NA		NA		NA		
18	60		10		200		

## 4.0 SAMPLING AND ANALYTICAL PROCEDURES

### 4.1 EVALUATION METHODS

The PFTIR evaluation was performed on the P001 Process Flare at varying operating scenarios as shown in Table 3-2. Control instrumentation was used to measure and vary the operating conditions and included:

- Flow meters and valves for natural gas, steam, and nitrogen streams;
- Process valves from C kettle for the 1,3-butadiene stream; and
- Panametrics for the stream from the seal tank.

A charge of hot water followed by liquid 1,3-butadiene was sent to a rubber kettle at the beginning of the evaluation. The jacket on the kettle heated the contents and caused the 1,3-butadiene to go into the vapor phase and flow to the Flare system. The vapors were sent to the Flare through the reflux vent valves for the low flows (~ 10 and 50 lbs/hr 1,3-butadiene flow) and through the four-inch vent header for the large flows (~300 and 600 lbs/hr 1,3-butadiene flow). The composition was measured by the Panametrics and flows were adjusted to get the desired 1,3-butadiene flow. Once the 1,3-butadiene flow was established, the nitrogen, natural gas, and steam flows were adjusted as necessary to get the desired NHVFG level.

During the evaluation, the INEOS ABS video camera recorded the plume in order to record any visible emissions (soot generation) from the plume during each scenario. Visible emissions can negatively impact the quality of data generated by the FTIR equipment and would prevent the ability to complete evaluations at specific operating scenarios. There were no test runs where visible emissions negatively impacted the results.

In addition, the INEOS ABS weather station was used to collect data (ambient temperature, relative humidity, ambient pressure, and wind speed) during the duration of each run for all the operating scenarios. This data was used to determine if a steam contribution factor should be applied to a particular test run.

The NHVFG value is the measure of enthalpy ( $\Delta H_f^{298}$ ) of the components that comprise the Flare Gas. Flare Gas is the sum of the entire stream measured by the Panametrics plus the supplemental natural gas sent to the Flare stack, plus the natural gas sent to the pilot burners, plus the steam determined to participate in the reaction zone. The density of the Flare Gas is determined by dividing the component molecular weight(s) by the molar volume at STP conditions (14.696 psia and 60°F, resulting in a STP molar volume of 379.48 ft<sup>3</sup>/lb-mol. References for these STP conditions include Perry's Handbook 8<sup>th</sup> Edition Table 1-4 Conversion Factors, as well as Crane's Technical Paper No. 410 on Flow of Fluids).

The molecular weight of each component is as follows:

The stream exiting the seal tank = Determined by the Panametrics  
Natural gas (based upon compositions from Duke Energy) = 16.40  
Steam = 18.015

The enthalpy of each component (using a reference point of 298.15 K for  $\Delta H_f$ ) is as follows:

1,3-butadiene (Perry's Handbook 8<sup>th</sup> Edition) = 19,160 BTU/lb

Natural Gas (NASA Polynomials; Duke Energy gas composition) = 915 BTU/scf

Steam (NASA Polynomials) = 131 BTU/lb (steam supplied to the tips at 458.7 K)

The NHVFG value was determined using the following equation and agreed upon by the EPA in August 2007:

$$(1,3\text{-butadiene flow [lbs/hr]} * 19,160 \text{ BTU/lb} + \text{natural gas (supplemental + pilots) flow [scfh]} * 915 \text{ BTU/scf} + \text{steam flow included [lbs/hr]} * 131 \text{ BTU/lb}) / (\text{lb-moles/hr of total Flare Gas} * 379.48 \text{ scf/lb-mole})$$

When exit velocities from the Flare are less than 75 feet per minute (ft/min), a steam contribution factor was applied when calculating the NHVFG and steam-to-Vent Gas ratio. It has been agreed to by the EPA and INEOS ABS during Consent Decree negotiations that at certain velocities only a portion of the steam participates in combustion with the Flare Gas just after the exit of the Flare tip. The EPA concluded that the steam contribution factor only includes 37.5% of the steam when exit velocities are below 75 ft/min. However, INEOS ABS believes that the appropriate exit velocity to apply a steam contribution factor is 100 ft/min. This exit velocity threshold is under appeal at the time of the writing of this report. The adjusted exit velocities using the steam contribution factor at exit velocities below 75 ft/min are shown in Tables 3-1 and 3-2.

The PFTIR evaluation measured the concentrations of CO<sub>2</sub>, CO, and THC which was used to estimate emissions of the Flare. The efficiency estimation was based on the following equation:

$$Eff = \frac{CO_2}{CO_2 + CO + THC}$$

where:

[CO<sub>2</sub>] = CO<sub>2</sub> concentration

[CO] = CO concentration

[THC] = THC concentration

NOTE: The above equation does not measure control or combustion efficiency. An actual efficiency calculation would include components that the FTIR equipment cannot detect (for example 1,3-butadiene, NO<sub>x</sub>, H<sub>2</sub>, etc.)

In order to complete these calculations the background concentrations of CO, CO<sub>2</sub>, and HC must be compensated. The procedure used to make these compensations is documented in "Combustion Efficiency of Flares" by Pohl, John H., Lee, Joannes, Payne, Roy and Tichenor, Bruce A., *Combustion Science and Technology*, (1986), 50:4, pp. 217 – 231 and in "Evaluation of the Efficiency of Industrial Flares: Test Results", EPA document EPA-600/2-84-095.

IMACC provided the following calculations that were used to estimate errors in efficiency with respect to each term in the PFTIR measurements. The variation in efficiency with each term is given by its partial derivative with respect to each term. This gives:

$$\frac{\delta Eff}{\delta CO} \Delta(CO) = \frac{-CO_2}{(CO_2 + CO + THC)^2} \Delta(CO)$$

$$\frac{\delta Eff}{\delta THC} \Delta(THC) = \frac{-CO_2}{(CO_2 + CO + THC)^2} \Delta(THC)$$

$$\frac{\delta Eff}{\delta CO_2} \Delta(CO_2) = \frac{(CO + THC)}{(CO_2 + CO + THC)^2} \Delta(CO_2)$$

In each term the  $\Delta(x)$  is the error for compound x. Once these terms are evaluated the total error in the efficiency is given by:

$$\sqrt{\left(\frac{\delta Eff}{\delta CO} \Delta CO\right)^2 + \left(\frac{\delta Eff}{\delta CO_2} \Delta CO_2\right)^2 + \left(\frac{\delta Eff}{\delta THC} \Delta THC\right)^2}$$

The efficiency as estimated by the PFTIR method was determined using the Classical Least Square method of analysis to two significant figures.

Measurements from the FTIR equipment were taken every second and averaged over a five minute period; an efficiency value was generated every five minutes. The samples taken at each operating scenario were averaged to get a representative efficiency for that particular operating scenario.

Anomalies in the data were evaluated before inclusion or exclusion in any analysis, e.g. efficiency values not consistent with other efficiency values in that test run. All of the concentration data reported by the PFTIR analysis has corresponding error measurements. These error values reflect how well the software is able to match the features in the infrared spectrum. By subtracting the scaled reference from the data, this ideally would result in a straight line. Deviations reflect inaccuracy in the measurement. The error bar is a root mean squared residual that can be converted to concentrations using library reference standards. If the data is not at least 2.5 times the value then the data is suspect. For example, IMACC stated that the measurement reported for  $C_3H_6$  concentration of 9.393 ppm on 11/03/09 at 10:46:47 AM is suspect because this value is less than 2.5 times the reported error for that measurement of 4.518 ppm.

IMACC also provided data that estimated the actual temperature at the point of the FTIR focus. This data was used to assess how successful the technicians were in keeping the point of focus of the FTIR sensor at the optimum location in the plume. The calculations employed by the FTIR computer assume the sensor is measuring the plume at 200°C. Therefore, deviations of that temperature from 200°C (used as a basis for the PFTIR analysis) lead to increased magnitude of error in the PFTIR test results. These temperature estimates are located in Appendix C.

## 4.2 PROCESS DATA

During the evaluation, the following parameters were measured and/or calculated using the existing steam and natural gas flow meters and the Panametrics and stored in the facility's data historian:

- Vent Gas sent to the Flare (in lbs/hr)
- 1,3-butadiene in the Vent Gas sent to the Flare (in lbs/hr)
- Steam flow sent to the Flare (in lbs/hr)
- Net Heating Value of the Vent Gas sent to the Flare (in BTU/scf)
- Natural gas in the vent stream sent to the Flare (in scfh)
- Steam-to-Vent Gas ratio
- NHVFG (in BTU/scf)

In addition, meteorological data and video of the Flare flame were collected and stored on the facility's computer network.

Copies of the monitoring parameters and meteorological data are included in Appendix D.

## 5.0 QA/QC ACTIVITIES

IMACC calibrated the PFTIR equipment prior to and at the end of each evaluation day. Calibration of the FTIR equipment included instrumental radiance calibration (calibration with a cold target), radiance calibration (calibration with an infrared black body source), and background radiance calibration (sky background). QA/QC audit data sheets are included in Appendix E.

The calibration parameters for the Panametrics (signal strength, etc.) was verified as being within proper ranges prior to, during, and after the PFTIR test period. During discussions between EPA and INEOS ABS concerning the methods used to determine 1,3-butadiene flow rates from the Panametrics data, a couple of problems were identified with the use of the Panametrics data that has led to a re-evaluation of the algorithm previously agreed to by INEOS ABS and the EPA in August 2007. This algorithm is being revised to address these problems. The revisions include development of logic to compensate for errors in the Panametrics estimate of Vent Gas molecular weight due to limitations in detecting nitrogen and the use of an assumed (i.e., constant) nitrogen concentration. GE Sensing has provided INEOS ABS an algorithm that can be used to estimate the errors in molecular weight measurements due to the use of an incorrect assumption for nitrogen composition in the stream. INEOS ABS is using this algorithm to develop a separate algorithm to correct the errors in the originally measured molecular weight. The INEOS ABS algorithm is currently being tested and INEOS ABS expects to complete corrections to the stream composition calculations for the period of the PFTIR by August 6, 2010.

Supplemental natural gas is metered via a thermal gas meter directly into the Flare base. Prior to the PFTIR tests a secondary thermal gas meter was installed in this line. This secondary gas meter indicated a higher flow rate than the original meter at most flow conditions. After the PFTIR testing was completed the original thermal gas meter was removed and sent back to the factory for recalibration. While that meter was out, an additional gas meter of a different design (mass flow meter) was installed in place of the original thermal gas meter. This meter agreed more closely with the newer (secondary) thermal gas meter. Therefore, the supplemental gas flow measurement from the secondary gas meter is considered to be the more accurate of the two meters in use during the PFTIR test period and its values are used in the calculations.

Steam flow to the Flare tip is metered via a vortex shedding meter. Prior to the PFTIR tests a secondary vortex meter was installed in the steam line. The readings from the secondary and the primary steam flow meters agree, therefore the primary steam flow indication is considered accurate for the PFTIR evaluation period.