

Completion of Final Report and Gas Analysis for a Biomass Gasifier

Submitted in partial fulfillment of Contract 55018a

of the Western Regional Biomass Energy Program

Contract beginning date: September 30, 1999

Contract ending date: December 31, 2001

Joe D. Craig, Principal Investigator

Cratech, Inc.

P.O. Box 70

Tahoka, Texas 79373

(806) 327-5220

Table of Contents

List of Figures..... ii

List of Tables..... ii

ABSTRACT..... iii

EXECUTIVE SUMMARY..... iiiii

1. INTRODUCTION..... 1

2. PROJECT SUMMARY..... 2

3. TASKS..... 3

1. Task 1: Report on Phase 2 and Phase 3 Progress Under Contract 55018. 3-5

2. Task 2: Gas Test Plan..... 6

3. Task 3: Gasifier Operation and Gas Sampling..... 6-13

4. Task 4: Results and Conclusions of the Gas Testing..... 14

1. Results..... 14

2. Conclusions..... 15

REFERENCES..... 15

ACKNOWLEDGEMENTS..... 15

APPENDIX: Biomass Gas Sampling and Analysis..... 16

List of Figures

Figure 1. Cratech Phase 2 Gasification System.....	2
Figure 2. Completed Phase 2 Gasification System: Feeder End View.....	3
Figure 3. Completed Phase 2 Gasification System: Filter End View.....	4
Figure 4. Performance of the Gasification System during One of the Commissioning Runs.....	5
Figure 5. Feeding Biomass into Process.....	7
Figure 6. Flaring Produced Gas.....	7
Figure 7. Wood Used for Fuel.....	8
Figure 8. Cotton Gin Trash Used for Fuel.....	8
Figure 9. Gas Chromatograph in Operation.....	9
Figure 10. Particle Filter.....	9
Figure 11. Impinger Data Collection.....	9
Figure 12. Set of Sampling Impingers.....	9
Figure 13. Gasification Process Performance.....	11
Figure 14. Composition of the Produced Gas as Measured by the CEM.....	12
Figure 15. Composition of the Produced Gas as Measured by the CG.....	13

List of Tables

Table 1. Proximate, Ultimate and Mineral Analysis of the Wood Used During Runs..	7
Table 2. Proximate, Ultimate and Mineral Analysis of the Cotton Gin Trash Used During Runs.....	8
Table 3. Summary of Gas Analysis.....	14

ABSTRACT

A new generation of small-scale (less than 20 MWe) biomass fueled power plants are being developed based on a gas turbine prime mover. These power plants are expected to increase the efficiency and lower the cost of generating power from fuels such as wood.

The development of this technology is being carried out in three phases. This report covers much of the phase 2 development effort. The goal of phase 2 is to complete a gasification system capable of fueling a 1 MWe gas turbine engine. This report includes description of the pressurized fluidized bed gasification system, the general performance of the system, the gas molecular content and the solid particle and alkali metal concentration of the produced gas.

Introduction

There is a great desire by many individuals and entities worldwide to convert the energy contained in indigenous biomass resources to useful forms of fuel and power at a cost that is competitive with non-renewable energy resources and with a responsible steward's view toward the environment. This is an immense and exciting challenge. Without further elaboration, Cratech's sole focus is to develop processes and products to meet this challenge.

There are several technical challenges to overcome before economical small scale

(1 to 20 MWe) biomass fueled power plants can become commercial reality. Figure 1 shows a schematic of a biomass-fueled integrated-gasifier gas turbine (biomass fueled IGGT) power plant. This is the path Cratech has chosen to provide the enabling technology to meet this market need. The technology is being developed in 3 phases. These phases are:

Phase 1: Biomass feed rate of 500 kg per hour at 2 bara (2 bar absolute) pressure

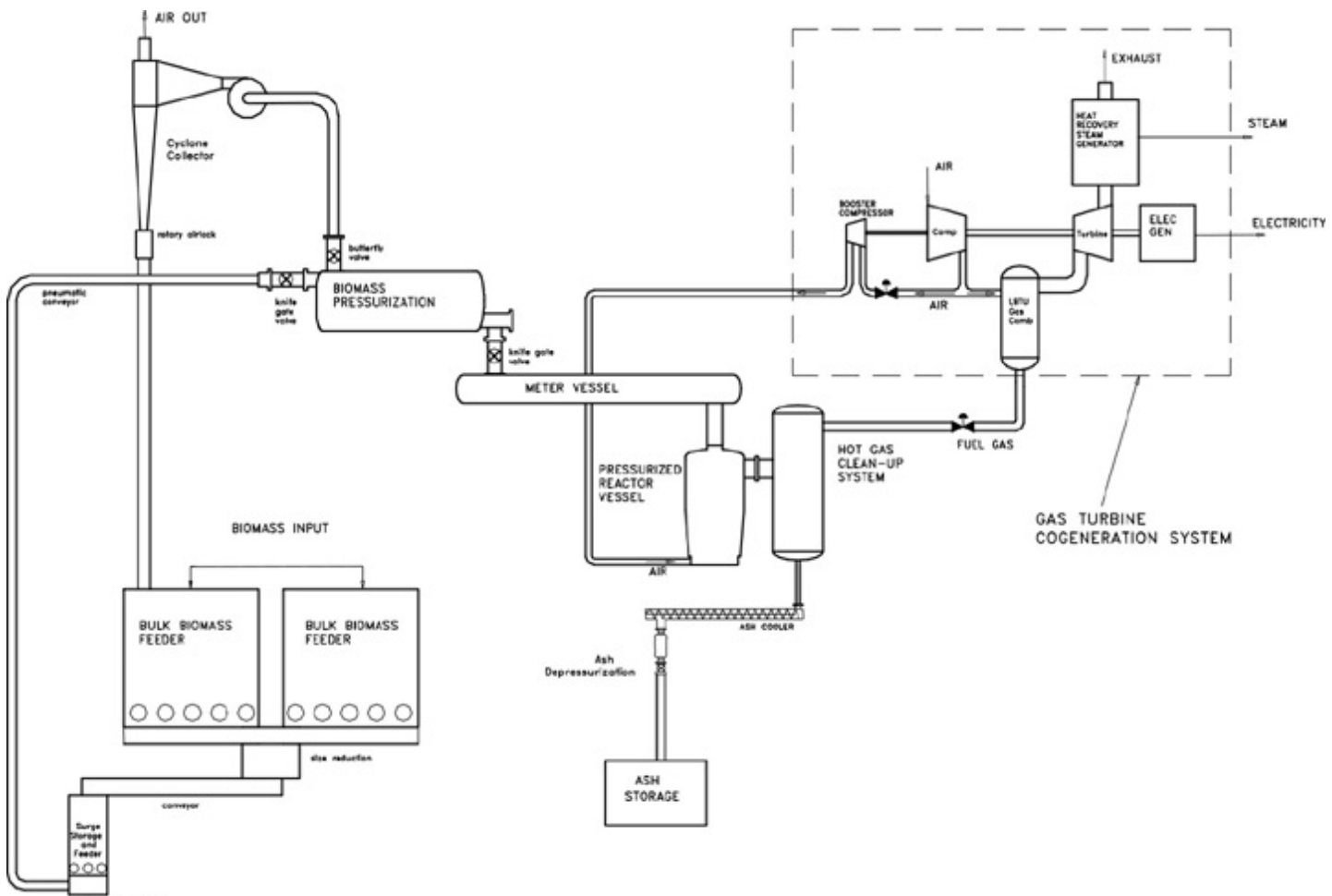
including a slipstream flow hot gas cleanup system.

Phase 2: Biomass feed rate of 1000 kg per hour at 10 bara pressure including a full

flow hot gas cleanup system.

Phase 3: Integrate the phase 2 system with a modified gas turbine engine.

Phase 1 of this development program is complete and is reported in (Craig, 1996). This report covers the progress made towards completion of Phases 2 and 3.



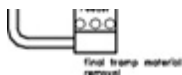


Figure 1. Cratech Phase 2 Gasification System

PROJECT SUMMARY

Design and construction of the phase 2 gasification system was completed. A series of operating runs were made to commission the system. Following the commissioning runs the system was declared ready for performance and gas emissions testing.

A gas test plan was prepared in cooperation with the US EPA. During June 2001 the gas test plan was completed. The system was operated for a total of 65 hours to complete the test plan including one continuous run of 45 hours.

The system was operated at a pressure of 2 bara at a temperature of 760° C using two different fuels. The performance parameters of the system were recorded. The gas produced was sampled for molecular composition and ash particulate and alkali metal concentration.

In addition, a study was completed to select a favorable site for the first commercial demonstration (beta test site) of the Cratech biomass power process. Work was also carried out towards completing phase 3. A gas turbine was procured and modifications were begun but further progress was put on hold due to lack of funding.

3.0 Tasks

Following are reports on each of the completed tasks.

3.1 Task 1: Report on phase 2 and phase 3 progress made under contract 55018

Construction of the phase 2 gasification system was completed and a series of low pressure (2 bara) commissioning runs were conducted beginning in April, 2000. A schematic of the system is shown in figure 1. Photos of the completed gasification system are shown in figures 2 and 3.



Figure 2. Completed Phase 2 Gasification System: Feeder End View



Figure 3. Completed Phase 2 Gasification System: Filter End View

The phase 2 system is rated to gasify 1000 kg/hr of biomass at a temperature of 760 °C and a pressure of 14 bara. The system was not operated at a pressure greater than 2 bara because the rated pressure capacity of the air compressor was limited. Following biomass gasification in the fluidized bed reactor the produced gas is processed through a hot gas filter at the given temperature and pressure. The commissioning run time totaled 63 hours. The gasification system was declared ready for a long term (up to 100 hour) performance and gas analysis run. Wood was used for fuel during the commissioning runs (see table 1 and figure 7).

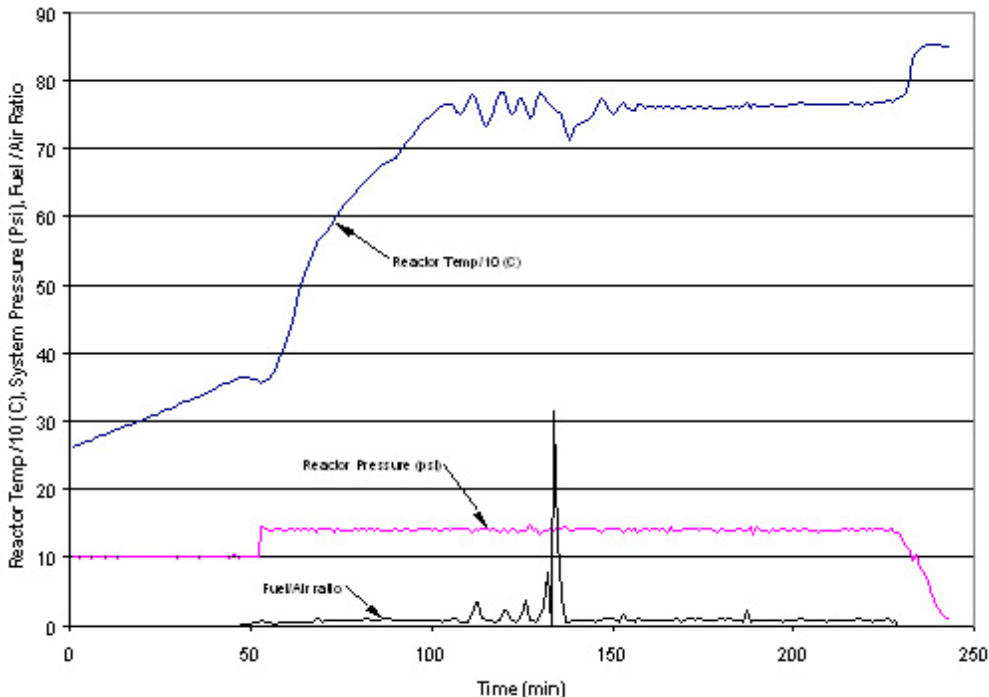


Figure 4. Performance of the Gasification System During One of the Commissioning Runs

Phase 3 included procurement and modification of a 1 MWe gas turbine genset to be fueled with the produced gas. We were not able to procure a 1 MWe gas turbine genset; however, we did obtain a 200 kWe gas turbine genset. The gas turbine genset had been in storage for

several years and its enclosure was in poor shape. The turbine engine itself appeared operable and in fairly good working order. The gas turbine unit was removed from its old enclosure, cleaned and repaired. It was then successfully operated with diesel fuel. A new enclosure for the turbine was designed and constructed. A new combustor was designed for the turbine so that it could be fueled with the produced gas. A report on this work was issued (Craig, 2000). Funding became insufficient to complete all work required on the turbine genset unit. Work on the unit was placed on hold.

A booster compressor will eventually be required to complete the biomass power plant. This compressor increases the pressure of the air supplied from the gas turbine engine to the gasification system (refer to figure 1, component labeled "booster compressor"). This is a major component required to properly integrate the phase 2 gasification system with the phase 3 gas turbine generator. The design and selection of this compressor was a requirement of the original contract 55018 (task 4). There were several possible choices for this compressor. The final choice was a compressor manufactured by Sundyne Corp of Arvada, Colorado.

3.2 Task 2: Gas Test Plan

In cooperation with the US EPA a gas test plan was prepared to test the gas produced by the phase 2 gasification system. The test plan is entitled, "**Biomass Gas Sampling and Analysis**" and is contained in the appendix.

3.3 Task 3: Gasifier Operation and Gas Sampling

The combined methods 5 and 29 gas sampling trains were prepared and set up to sample the produced gas over a maximum time period of 100 hours. The gas chromatograph and continuous emissions monitoring instruments were also set up to sample gases for a maximum time run of 100 hours.

Wood was fed to the gasifier from June 01, 2001 12:00 to June 03, 2001 00:00 (see figure 7 and table 1). Cotton gin trash (CGT) was fed to the gasifier from June 03, 2001 00:00 to June 03, 2001 9:00 (see figure 8 and table 2). This was a continuous test run of 45 hours.

Figure 5 shows the biomass being loaded into the bulk feed hopper. Figure 6 is a photo of the resulting produced gas. The molecular composition of the gas was measured by the use of continuous emissions monitors and a gas chromatograph, see figure 9. The gas was measured for its solid particle content by use of modified EPA method 5. Method 5 uses quartz thimble filters to capture any particles entrained in the gas, see figure 10. The gas was analyzed for its alkali metal content by the use of method 29, see figures 11 and 12.



Figure 5. Feeding Biomass into Process



Figure 6 . Flaring Produced Gas

Table 1. Proximate, Ultimate and Mineral Analysis of the Wood Used During Runs (see figure 7 for photo)

Proximate Analysis (as received basis)

component	weight %
moisture	8.9
ash	0.59
volatiles	37.6
fixed carbon	52.9
Heating Value	20.00 mJ/kg

Ultimate Analysis (dry basis)

component	weight %
carbon	48.2
hydrogen	5.4
nitrogen	0.05
sulfur	0.01
ash	0.59
oxygen (diff)	36.92

Mineral Analysis

Mineral	weight %
SiO ₂	48.5
Al ₂ O ₃	28.0
TiO ₂	1.6
Fe ₂ O ₃	1.7
CaO	12.3
MgO	1.7
K ₂ O	3.6
Na ₂ O	0.5

Mineral	weight %
SO ₃	0.70
P ₂ O ₅	1.03
SrO	0.11
BaO	0.26
Mn ₃ O ₄	0.10



Figure 7. Wood used for fuel, see also table 1.



Figure 8. Cotton Gin Trash used for fuel, see also table 2.

Table 2. Proximate, Ultimate and Mineral Analysis of the Cotton Gin Trash (CGT) Used During Runs (see figure 8 for photo)

Proximate Analysis (as received basis)

component	weight %
moisture	9.0
ash	7.75
volatiles	45.7
fixed carbon	45.81
Heating Value	19.98 mJ/kg

Ultimate Analysis (dry basis)

component	weight %
carbon	45.28
hydrogen	5.14
nitrogen	1.58
sulfur	0.40
ash	8.52
oxygen (diff)	38.54

Mineral Analysis

Mineral	weight %
SiO ₂	7.20
Al ₂ O ₃	6.94
TiO ₂	0.11

Fe ₂ O ₃	0.40
CaO	20.74
MgO	10.31
K ₂ O	35.64
Na ₂ O	1.27

Mineral	weight %
SO ₃	12.64
P ₂ O ₅	4.44
SrO	0.13
BaO	0.11
Mn ₃ O ₄	0.07



Figure 9. Gas Chromatograph in Operation.



Figure 10. Particle filter



Figure 11. Impinger data collection



Figure 12. Set of Sampling Impingers

The charts shown in figure 13 are the recorded general performance of the gasification system. Figure 14 are two charts showing the data recorded by the continuous emissions monitors. Figure 15 is a chart showing the data recorded by the gas chromatograph. All data given is for the 45 hour continuous run.

The continuous run was interrupted by the failure of the ash rotary airlock to maintain a constant pressure. This airlock is part of the component labeled "ash depressurization" in figure 1.

In an attempt to gather more data additional runs were made. This was possible by manually starting and stopping the rotary airlock. When the airlock was not rotating the ash would build up and the pressure in the system increased. An additional 20 hours of short run operation was achieved in this manner; however, no additional useful gas sampling data was achieved during these short term runs. The rotary airlock began to fail completely to maintain system pressure. The test runs were terminated.

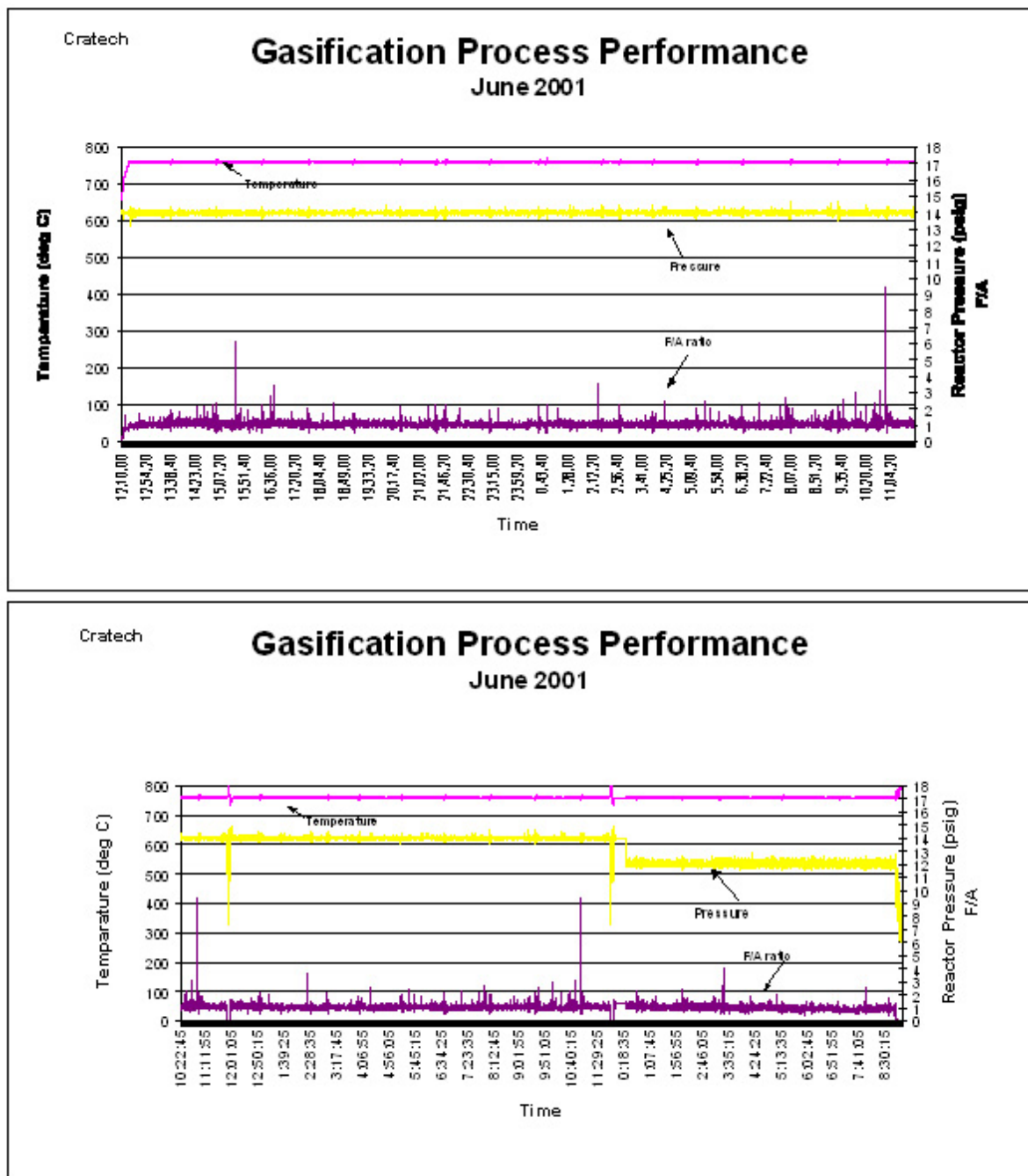


Figure 13. Gasification Process Performance (top chart is first half of run, lower chart is second half of run)

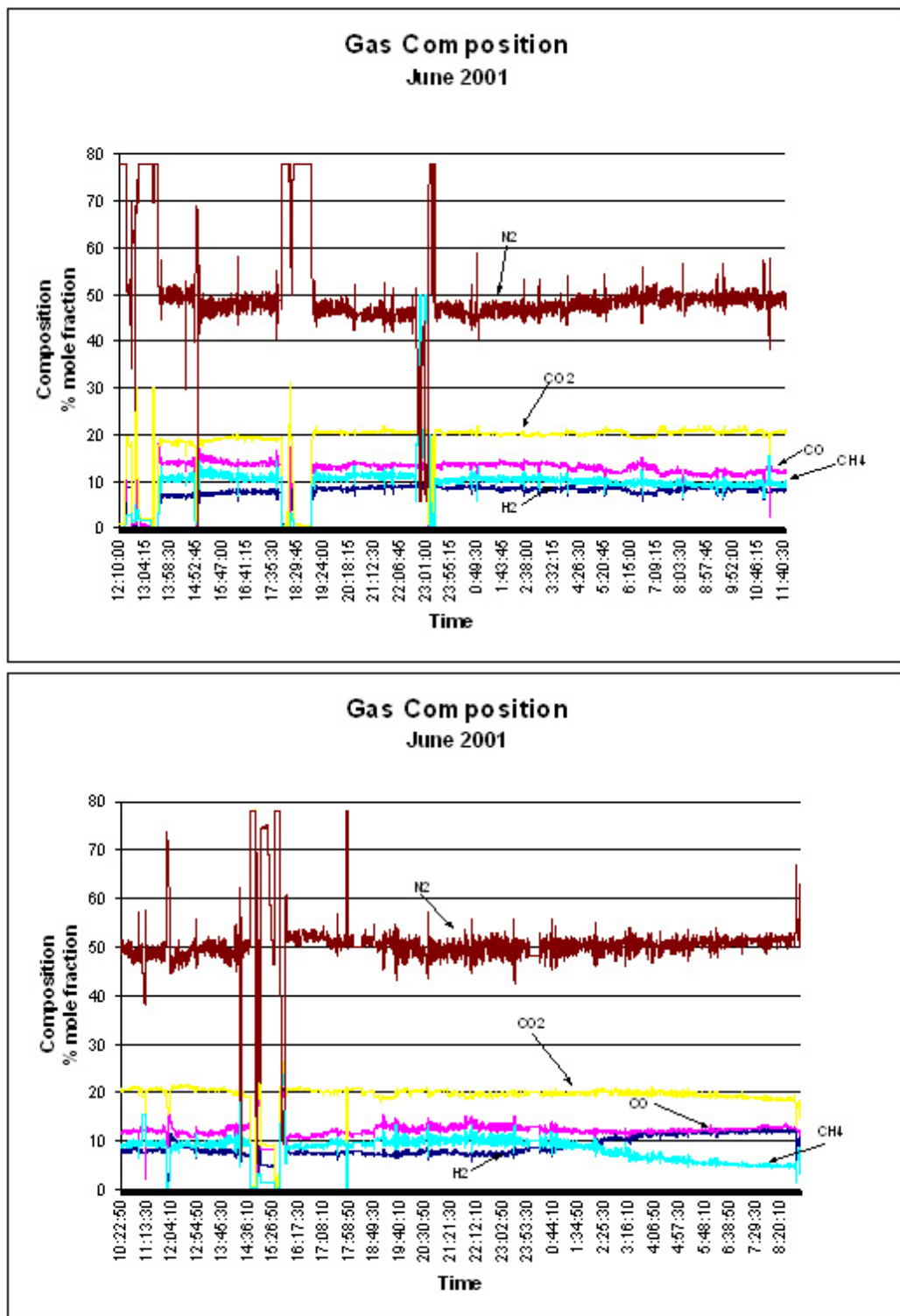


Figure 14. Composition of the Produced Gas as measured by the CEM (top chart is first half of run, lower chart is second half of run)

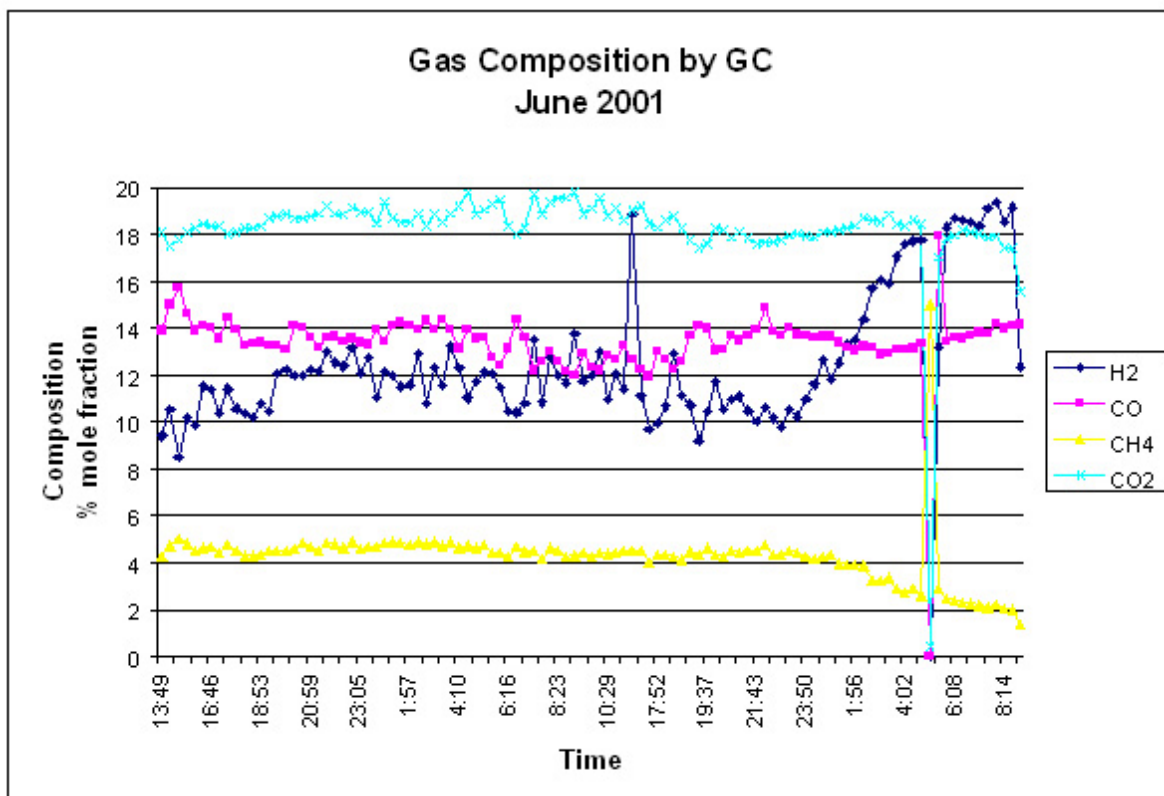


Figure 15. Composition of the Produced Gas as measured by the GC.

3.4 Task 4: Results and Conclusion of the Gas Testing

The following are the analysis of the results of the testing and some conclusions drawn from analysis of the results.

3.4.1 Results

Table 3 is a summary giving the solid particle concentration, alkali metal concentration and gas composition for the three wood sampling runs, 2A, 3A and 3C and the two CGT runs 4A-2 and 4B.

Table 3. Summary of Gas Analysis

Run	Date	Particulate*	Na*	K*	H2%	CO%	CO2%	CH4%	O2%
2A	6/1/01	99.9	0.51	3.95	9.96	11.41	16.8	8.98	0.23
3A	6/2/01	-	0.63	0.56	12.26	12.82	20.28	9.97	0.26
3C	6/2/01	302.5***	0.4	2.4	11.14	10.9	18.56	8.21	0.75
4A-2	6/3/01	-	28.6	2.2	17.74	12.57	19.07	5.30	0
4B	6/4/01	20.6****	1.9	9.4	12.85	10.99	17.68	5.55	0.06
* - ppmw									
*** - average of 3A & 3C									
**** - average of 4A-2 & 4B									

Sampling the pressurized hot gas for its molecular composition by GC and CEM with the method described proved to be adequate. Cooling

the gas for use by the instruments was not difficult except for tar condensation. The cooling apparatus had to be constantly monitored and cleaned of tars to maintain gas sampling flow.

The EPA modified method 5 was very difficult to use for sampling the hot pressurized gas. The gas sampling filter and the impinges were operated under the gas sampling pressure of 2 bar (abs) and a temperature of 760 °C. The primary difficulty was assuring that the temperature of the sampling filter was maintained at the same temperature as the gas being sampled and under pressure. Following each run the sample filters seemed to be coated with a black film on the outer and inner surface (see figure 10). The filter is white in color and should remain close to this color when removed. The only agreed upon explanation for this phenomenon is that heating of the sample filter may have caused the tar vapors passing through the filter to become solidified. This problem lends considerable uncertainty to the resulting solid particle concentration data as reported in table 3. Unfortunately this also taints the reported alkali metal concentration results as shown in table 3.

3.4.2 Conclusions

1. The performance of the gasification system is stable when fed wood or cotton gin trash.
2. The currently installed ash rotary airlock is not suitable for this application.
3. The molecular gas composition produced by this system is typical for air-blown gasification.
4. Changes to the gas sampling train are required before an accurate measurement of the solid particle concentration can be determined.
5. Changes to the gas sampling train will be required before an accurate measurement of the alkali metal concentration can be determined.

REFERENCES

Craig, Joe D. Development of a Small Scale Biomass-Fueled Integrated-Gasifier Gas Turbine Power Plant: Phase 1. Western Regional Biomass Energy Program, Golden, CO. September 24, 1996.

Craig, Joe D. Development of a Small Scale Biomass-Fueled Integrated-Gasifier Gas Turbine Power Plant: Phase 2 and 3. Final Report for the Western Regional Biomass Energy Program. Grant DE-FC48-97WA13475 Golden, CO. February 1, 2000.

Craig, Joe D. Small Scale Biomass Power Plant Site Evaluation Within the State of Texas. General Services Commission State Energy Conservation Office of the State of Texas. Contract C9012. May 21, 1999.

ACKNOWLEDGEMENTS

U.S. Department of Energy, Western Regional Biomass Energy Program

U.S. Environmental Protection Agency, National Risk Management Research Laboratory

State of Texas, General Services Commission, State Energy Conservation Office.

Texas Tech University

APPENDIX: Biomass Gas Sampling and Analysis

Biomass Gas Sampling and Analysis

Quality Assurance Project Plan

Category IV/Basic Research

U.S. Environmental Protection Agency

Air Pollution Prevention and Control Division

March 2001

Contract No. 68-C99-201

Work Assignment No. 2-032

Project No. RN992012.0032

Prepared for:

U.S. Environmental Protection Agency

Air Pollution Prevention and Control Division

Atmospheric Protection Branch

Research Triangle Park, NC 27711



P.O. Box 13109

Research Triangle Park

North Carolina 27709

QAPP

March 2001

Biomass Gas Sampling and Analysis

Quality Assurance Project Plan

Category IV/Basic Research

U.S. Environmental Protection Agency

Air Pollution Prevention and Control Division

March 2001

Contract No. 68-C99-201

Work Assignment No. 2-032

Project No. RN992012.0032

Prepared for:

U.S. Environmental Protection Agency

Air Pollution Prevention and Control Division

Atmospheric Protection Branch

Research Triangle Park, NC 27711

EPA

Work Assignment Manager Carol Purvis Date

ARCADIS Geraghty & Miller

Work Assignment Leader Russ Clayton Date

EPA

QA Representative Paul Groff Date

ARCADIS Geraghty & Miller QA Officer Laura Beach Date

CONTENTS

-

Signature Page

Section - Page

1.0 PROJECT OBJECTIVES AND ORGANIZATION 1 – 1

1.1 Background 1 – 1

1.2 Project Objectives 1 – 1

1.3 Project Management 1 – 1

2.0 EXPERIMENTAL APPROACH 2 – 1

2.1 Test Facilities 2 – 1

3.0 SAMPLING AND ANALYSIS PROCEDURES 3 – 1

3.1 Sampling Methods 3 – 1

3.1.1 Particulate and Metals 3 – 1

3.1.2 GC/TCD Sampling 3 – 2

3.1.3 Continuous Emission Monitors 3 – 2

3.2 Analytical Methods 3 – 4

3.2.1 Particulate 3 – 4

3.2.2 Metals 3 – 4

4.0 QA/QC CHECKS 4 – 1

4.1 Bias, Precision and Completeness 4 – 1

4.2 Comparability 4 – 1

4.3 Internal QC Checks 4 – 2

4.4 Corrective Action 4 – 2

5.0 DATA REDUCTION, VALIDATION, AND REPORTING 5 – 1

5.1 General Principles 5 – 1

5.2 Data Reduction 5 – 1

3. Data Validation 5 – 1

4. Data Reporting 5 – 1

6.0 References 6 – 1

List of Figures Section-Page

Figure 1-1. Project Organization 1 – 2

Figure 2-1. Test Facility 2 – 2

List of Tables Section-Page

Table 4-1. Data Quality Indicator Goals for Critical Measurements 4 – 3

Appendices

Appendix A. Standard Methods

1.0 PROJECT OBJECTIVES AND ORGANIZATION

1.1 Background

The EPA/APPCD has an interest in small-scale, biomass-fueled power plants, in particular, the molar composition and contaminants in a

biomass syngas produced by a pressurized, fluid-bed gasifier and cleaned with a hot gas filter system designed to fuel a gas turbine. The gas compatibility of the syngas with a gas turbine is critical in the long-term operation of such a power plant.

1.2 Project Objectives

ARCADIS will conduct sampling and monitoring to characterize the biomass-produced syngas at a pilot system owned and operated by Cratech in Tahoka, Texas. The following tasks will be performed:

- Furnish gas sampling and analysis equipment
- Perform 8-hour shakedown test at facility
- Perform 100-hour continuous run for sampling
- Collect and analyze samples

The primary constituents of interest are particulate, sodium and potassium concentration, and process gas concentration (CO, CO₂, O₂, NO_x, and methane).

1.3 Project Management

Carol Purvis is the EPA/APPCD Work Assignment Manager for this project. ARCADIS personnel responsible for carrying out the project are shown in the project organization chart, Figure 1-1. Kevin Bruce is the Project Manager for the APPCD On-Site Laboratory Support contract. Russ Clayton is the Work Assignment Leader for ARCADIS with overall responsibility for task coordination, sampling supervision, data generation and analysis, and corrective action. Mr. Clayton will also be ultimately responsible for reviewing all necessary documents (test plan, safety plans, monthly progress reports, journal publications, and final reports). Gene Stephenson is the Sampling Engineer for this project. He will be responsible for sample collection and recovery, data generation, and report preparation. Mike Bowling will be a member of the field crew to assist Mr. Stephenson in testing and sampling. Laura Beach, ARCADIS QA Manager, will provide QA support including performance evaluation materials requested by EPA, QAPP review, and necessary internal audits. Category IV projects are not routinely audited unless requested by the EPA WA Manager. Jerry Revis, ARCADIS Safety Officer, will provide safety oversight of the project.

Responsibility for producing quality data is shared by all staff members, and each member shares the responsibility for reporting data quality problems to the WA Manager or the QA Manager. Overall data quality will be continuously assessed in the field by the Sampling Engineer and data are reported by the ARCADIS Work Assignment Leader and the EPA Work Assignment Manager. Joe Craig of Cratech will be responsible for facility preparation and operation under subcontract.

Figure 1-1. Project Organization

2.0 EXPERIMENTAL APPROACH

2.1 Test Facilities

A pilot system located in Tahoka, Texas, owned by Cratech, will be the host for this testing. The Cratech Pressurized Fluidized-Bed Gasification System is shown in Figure 2-1. The Bulk Biomass Feeder is a live-bottom screw feed hopper that is filled with biomass material using a wheeled loader. From this atmospheric pressure hopper, the biomass material is conveyed pneumatically to the Biomass Pressurization Vessel (BPV). The BPV, by use of knife gate valves, carries material batches across the barrier between atmospheric and fluidized-bed operating pressures. From the BPV, material is delivered to the top of the Meter Vessel (MV). The MV, a pressurized gravimetric feeder, under microprocessor control, meters biomass material into the top of the Reactor Vessel. The Reactor Vessel is an air-blown fluidized-bed reactor operating at up to 103 kPa and approximately 740 °C. It exhausts into the Receiver Vessel and Ash Cooling and Removal System for large particle and ash depressurization and removal. The Receiver Vessel is essentially a pressure surge tank and manifold which conveys most of the product gas (~95%) to the Flare through the Hot Gas Pressure Regulator Valve (which is the primary system pressure regulator); the remaining 5% is conveyed to the Hot-Gas Filter Vessel (HGFV) for fine particle removal. The HGFV uses sintered metal filters to remove all particles 2.8 •m in diameter and larger. The product gas volume and quality are measured in the Gas Flare Proximate Calorimeter. The particulate gas train tap will be between the HGFV and the calorimeter. The Control and Data Acquisition System uses ABB Kent-Taylor Modcell 2000 microprocessor controllers with a PC for operator interface.

3.0 SAMPLING AND ANALYSIS PROCEDURES

Sampling will be conducted at a bypass pipe between the exit of the gasifier filter vessel and the flare. EPA Methods 5 and 29 will be combined into a single sampling train for this project for the determination of particulate and metals (sodium and potassium). GC sampling will be according to standard on-line GC methodology as used at the EPA/ERC in RTP. CEM measurements for CO₂/O₂, NO_x, and CO will be conducted according to the respective EPA reference methods (3A, 7E, and 10).

Samples will be withdrawn isokinetically from the source using a combined EPA Method 5 and EPA Method 29 sampling train. The metals of interest are sodium and potassium. The EPA Method 5 portion of the sampling train will quantify total particulate.

The combined sampling train will consist of a stainless steel nozzle, a stainless steel filter holder with a thimble filter for particulate capture, seven chilled impingers, and a metering console. The particulate filter will be a Whatman 19 x 90 mm quartz thimble filter. The first and second impingers will each contain 100 mL of deionized, distilled water, the third impinger will remain empty, a second 3 in. quartz filter will be installed between the third and fourth impingers to help prevent tars (which are expected to be removed by the first three impingers) from contaminating the metals reagents, the fourth and fifth impingers each will contain 100 mL of 5% nitric acid (HNO_3)/10% hydrogen peroxide (H_2O_2), the sixth impinger will remain empty, and the seventh impinger will contain silica gel. Each impinger will be weighed prior to sample train assembly. Each run will be a minimum of 2 hours in duration with a minimum sample volume of 60 dsf³. A sample pump is not necessary due to the reactor's being under high positive pressure. The stainless steel nozzle is fixed semipermanently to the gasifier. It is rinsed with 0.1N HNO_3 and is not expected to

contribute sodium or potassium contamination to the samples.

Impingers will be monitored visually to determine the maximum run length possible without contamination of the metals reagents. Runs will be as long as possible (with changing impinger trains if necessary while keeping the same particulate filter) to catch as much particulate as possible due to the very low expected particulate loading (and correspondingly low and difficult-to-measure catch weight). In general terms, run lengths and filter changout times will be dictated by current conditions on site (i.e., decisions will have to be made in real time to maximize data quality).

Each impinger will be weighed to determine the post-test weight gain. The volume of water vapor condensed in the impingers and the volume of water vapor collected in the silica gel will be summed and entered into moisture content calculations.

A Teflon spatula and Teflon-coated tweezers will be used to remove the filter from the filter holder and place it in a 250 mL glass jar. The impinger reagents will be transferred to a 1000 mL glass jar.

The nozzle, probe, and front half of the filter holder will be rinsed with acetone into a 250 mL glass jar followed by rinsing with 100 mL of 0.1N HNO₃ into a second 250 mL glass jar. A Teflon probe brush will be used for cleaning the probe.

The back half of the filter holder and the first, second, and third impingers will be rinsed with 100 mL of 0.1N HNO₃ into the 1000 mL jar containing the HNO₃/H₂O₂ reagent, and the liquid level will be marked. Impinger samples and filters will be quantitatively transferred to labeled sample bottles and adequately stored until transfer to the commercial laboratory for analysis.

2. *GC/TCD Sampling*

Continuous emissions monitoring of five fixed gases (hydrogen, nitrogen, carbon monoxide, carbon dioxide, and methane) will be analyzed using a Hewlett Packard 5890 Gas Chromatograph (GC) equipped with a thermal conductivity detector (TCD). Positive effluent from the process stream will enter a moisture removal device prior to being routed through a 5 mL sample loop that is mounted on a six-port, two-position, electronically actuated valve. After a small equilibration time to allow standard/sample through the tubing and sample loop, the GC is started via an HP 3396 B integrator that uses a basic program to run the system. Once the GC system has started, time-programmed relays are used to equilibrate the sample loop prior to injection and also to open the 2-position valve which allows the 5 mL sample loop to enter the carrier gas stream. Constituents are separated due to each compound's unique size and diffusion characteristics through the Haysep DB30 material rather than typical boiling point and column affinity effects found in most GC analyses. The TCD measures the difference in thermal conductance between the carrier gas and the sample analytes. Samples can be analyzed every 15 minutes from the process stream.

GC calibration will consist of gases at three concentration points: zero %, ~80% of span, and ~ 40% of span. Calibration drift and system bias checks will be done twice daily.

3. *Continuous Emission Monitors*

Continuous emissions monitoring (CEM) will be conducted for carbon dioxide (CO₂), carbon monoxide (CO), oxygen (O₂), and nitrogen oxide (NO_x) concentrations. The sampling and analytical procedures for CO₂/O₂, NO_x, and CO will be in accordance with EPA Methods 3A, 7E, and 10, respectively.

The extractive monitors require that the effluent gas sample be conditioned to eliminate any possible interference (i.e., water vapor and/or particulate matter) before being transported and injected into each analyzer. All components of the sampling system that contact the sample are constructed of Type 316 stainless steel or Teflon. The outputs from the monitors are connected to a computerized data acquisition system (DAS) maintained by Cratech.

The CO₂, O₂, CO, CH₄, H₂, and NO_x sample collection system consists of a stainless-steel tap into the gasifier stream, isolation valve, a moisture removal trap, and out-of-stack secondary quartz particulate filter. All samples are routed through a distribution manifold board for delivery to the analyzers. The configuration of the sampling system allows for the injection of calibration gases directly to the analyzers or through the sampling system. A sample pump is not

required due to sampling under pressure. All pretest and posttest calibration procedures are performed as outlined in the specific EPA methods. A system leak check will be performed daily by closing the isolation valve and pressurizing the line. Any loss of pressure will be repaired prior to sampling. The operation of each analyzer and the DAS is described in the following subsections.

Horiba PIR-2000 CO/CO₂ Analyzer and Servomex Model 1440C1 CO/CO₂/CH₄ Analyzers

This non-dispersive infrared analyzer (NDIR) automatically and continuously monitors the CO and CO₂ concentrations. The theory of operation is based on the principle that both gases have a unique absorption line spectrum in the IR region. The instrument consists of an IR light source, a chopper, a measuring cell, and a two-chamber sealed detector.

The IR light beam emitted by the source passes through a measuring cell that is filled with a continuously flowing gas sample. Before reaching the front chamber of the detector, the light beam is partially absorbed or attenuated by the gas species of interest in the cell. Both the front and rear chambers of the detector are filled with a reference gas. The difference in the amount of light absorbed between the front and rear chambers is dependent on the concentration of the gas species of interest within the measuring cell and creates a pressure differential between the two chambers. The pressure differential is then observed as gas flow by the microflow sensor located in the channel connecting the two chambers. The resulting AC signal from the sensor is rectified, amplified, and linearized into a DC voltage signal for output.

TECO Model 10 NO/NO_x Analyzer

This analyzer automatically and continuously determines the concentration of the nitric oxide (NO) or nitrogen oxides (NO_x) in the effluent. The analytical technique is chemiluminescence. The sample is routed through a converter where the NO₂ is dissociated to form NO. The sample is then passed through a reaction chamber where the NO is quantitatively converted to NO₂ by gas-phase oxidation with molecular ozone produced within the analyzer. In this reaction, the NO₂ molecules are elevated to an electronically excited state and immediately revert to a non-excited ground state. This reversion is accompanied by the emission of photons, which impinge on a photomultiplier detector and generate a low-level DC current. The current is then amplified and used to drive a front panel meter and a data recorder. The NO_x concentration seen by the instrument includes the contributions of both the NO in the sample and the NO resulting from the dissociation of the NO₂ in the sample.

Beckman Model 755 O₂ Analyzer and Servomex Model 1440C1 O₂ Analyzer

This analyzer was designed specifically to measure O₂ in flue gas streams. It utilizes the magnetic susceptibility of O₂ by passing it through a strong, non-linear magnetic field. As it responds to the magnetic field, the O₂ displaces a detector located in the analyzer's measurement cell. The degree of displacement is proportional to the concentration of O₂ in the sample. An electrical circuit wired into the detector provides a signal output of 0 to 25% O₂.

MSA Thermatron Thermal Conductivity Analyzer

Operation of the Thermatron is based upon the effect of a gas sample upon the temperature of a heated wire filament. The filament is placed within a cylinder well of a cell block and is one arm of a Wheatstone bridge circuit. The filament is heated, and heat is lost to the cell walls at a rate depending upon the thermal conductivity of the gas. A change in concentration of the gas changes the rate of heat loss and upsets the bridge circuit, resulting in an analog signal proportional to the concentration of the component of interest.

Data Acquisition System

The DAS consists of an ABB process controller for data acquisition and a Pentium-based PC with a 5 GB hard drive for data storage. The software for data display and collection is Intellution. Data are collected from the various instruments (H₂, CO, CO₂, CH₄, NO_x, O₂, temperature of the sample gas, and rate of gas being sampled) via an ABB Modcell 2000 process control and data acquisition system. Data are collected from each channel at the rate of once per second and communicated to the PC. The data are stored in a format that can be imported into an Excel spreadsheet for data analysis and graphing. The DAS uses a DELL portable computer with a 20 MB hard disk and a strip chart recorder/analog-to-digital converter. In addition to providing an instantaneous display of analyzer responses, the DAS compiles the analyzer data collected once each second and averages them. The DAS integrates the real-time measurements and provides printouts of averaged emissions over any time period desired.

Continuous emission monitors will be operated for NO_x, CO, CO₂, O₂, CH₄, and H₂. The following QA/QC criteria will be implemented for NO_x, CO, CO₂, and O₂ as discussed in the applicable federal register methods. Instrument calibration/linearity will be required to be correct within 2% of full scale. Zero and calibration drift will be limited to 3% of full scale. Sampling system bias will be limited to 5% of full scale. These QA/QC criteria should be met in most cases.

CEM calibration will consist of gases at three concentration points: zero %, ~80% of span and ~ 40% of span. Calibration drift and system bias checks will be done twice daily.

3.2 Analytical Methods

1. *Particulate*

Samples will be analyzed by First Analytical Laboratories for particulate catch, sodium, and potassium. Analysis for particulate will be conducted according to EPA Method 5. The acetone rinse will be transferred to a tared weighing container and evaporated to dryness at ambient temperature and pressure. The filter will be transferred to a tared weighing container. Both containers will be desiccated for a minimum of 24 hours, weighed to a constant weight, and the results reported to the nearest 0.1 mg.

2. *Metals*

Sample preparation and analysis for potassium and sodium will be conducted as outlined in EPA Method 29.

The filter will be quartered and placed in a microwave pressure relief vessel. Concentrated HNO₃ (6 mL) and concentrated HF (4 mL) will be added to the vessel. Then it will be heated in a microwave oven until sorbent reflux is evident, and cooled at room temperature.

The acetone rinse residue will be redissolved with 10 mL of concentrated HNO₃ and added to the 0.1N HNO₃ probe rinse. This sample will then be rinsed into a beaker, using distilled water. The sample will be reduced to approximately 20 mL by heating on a hot plate and will then be transferred to a microwave oven and treated in the same manner as the filter. The resultant sample will be combined with the digested filter and will be designated "Sample Fraction 1." The sample will then be filtered and diluted to 300 mL (or other appropriate volume) and labeled "Analytical Fraction 1."

The impinger reagent volume will be determined and labeled "Sample Fraction 2." This sample will be digested using the microwave digestion procedure outlined in Section 8.3.4.2 of EPA Method 29 and will be labeled "Analytical Fraction 2." Analytical fractions 1 and 2 will be combined and analyzed for sodium and potassium using flame atomic absorption (FLAA) or graphite furnace atomic absorption (GFAA).

4.0 QA/QC CHECKS

4.1 Bias, Precision, and Completeness

The data quality indicator goals (DQIGs) are specific criteria used to quantify how well the collected data meet the appropriate data quality objectives. The DQIGs for bias, precision, and completeness (defined below for the critical measurements) are listed in Table 4-1.

Bias - The degree of agreement between an average measurement and an accepted reference or true value, expressed as a percentage of the reference or true value. Accuracy DQIG must include systematic errors associated with the sampling process.

$$\%Bias = \frac{AveragedMeasuredValues - KnownValue}{KnownValue} \times 100$$

Precision - Expressed as percent relative standard deviation (RSD), it can be determined using the formula:

$$RSD = \frac{\text{Standard Deviation of Replicate Measurements}}{\text{Average of Replicate Measurements}} \times 100$$

$$\% \text{Completeness} = \frac{\text{Amount of Valid Data Collected}}{\text{Intended Collectable Data}} \times 100$$

Completeness- Expressed as percent acceptable data collected,

using the expression:

Compliance with the DQIG for accuracy of the continuous emission monitors (CEMs) will be assessed by introducing a span gas through the entire sampling system on at least one occasion to allow for comparison between the measured value and the manufacturer's stated value. Precision of the CEM measurements will be assessed by calculating the relative standard deviation of replicate measurements of calibration gases of known concentration.

4.2 Comparability

Comparability evaluates the degree to which the collected data can be compared with measurements of similar parameters at similar locations or from similar systems. All test data that satisfy the data validation criteria will be compared consistently with the reference methods used or with the procedures and standard units derived and agreed upon during the finalization of this QAPP. The test data are representative of this unique experimental research site and are not directly comparable with other gas turbines.

4.3 Internal QC Checks

To ensure sampling reproducibility, replicate tests will be conducted as needed. All reagents will be of a reagent or pesticide grade. All information not recorded on standardized data sheets will be recorded in a bound project notebook. Samples generated in the field will be labeled. Integrity of the samples will be maintained by using proper chain-of-custody procedures.

A single field blank will be prepared for the EPA Method 5/29 (particulate/metals) train. It will be analyzed along with actual samples to access bias in train preparation and cleanup. The same applies to a reagent blank. Analytical laboratory QC samples will be treated according to EPA Methods 5 and 29. QC checks on the GC and CEMs will be treated according to EPA Method 6C.

4.4 Corrective Action

The predetermined limits for data acceptability are listed in Table 4-1. If these limits are exceeded, corrective action will be taken. This action may be a notation in the final report, recalibration of a particular instrument, a repeat analysis, or, in extreme cases, the repeat of an entire test. Therefore, the Work Assignment Leader's judgment will be important in corrective action initiation. In this judgement, the Work Assignment Leader will be guided by the relative importance of a given measurement to the overall test objectives, and the degree to which the data quality limits have been exceeded.

All staff members are responsible for reporting any identified QA problems to the Work Assignment Leader. The Work Assignment Leader will be responsible for approving and/or ensuring the performance of the corrective action.

TABLE 4-1. Data Quality Indicator Goals for Critical Measurements

Measurement	Test Method	Accuracy (% Bias)	Precision (%RSD/RPD)
Particulate	EPA Method 5	NA	10% (replicate lab analyses)
Metals (K, Na)	EPA Method 29	NA	10% (replicate lab analyses)
Product Gas Flow	Orifice Meter/Cratech	±5%	NA

Product Gas Moisture	EPA Method 5/29 (impingers)	±1% moisture	±5%
NO _x	CEM EPA Method 7E	±5 (of full scale)	±5 (of full scale)
O ₂	CEM EPA Method 3A	±5 (of full scale)	±5 (of full scale)
CO ₂	CEM EPA Method 3A	±5 (of full scale)	±5 (of full scale)
CO	CEM EPA Method 10	±5 (of full scale)	±5 (of full scale)
CH ₄	CEM	±5 (of full scale)	±5 (of full scale)
H ₂	CEM	±5 (of full scale)	±5 (of full scale)
H, N ₂ , CO/CO ₂	GC/TCD	±5 (of full scale)	±5 (of full scale)
Methane	GC/TCD	±5 (of full scale)	±5 (of full scale)

N₂ and CO co-efficients and may not be quantified.

Complete data for measurements in the 6

DATA REDUCTION, VALIDATION AND REPORTING

1. General Principles

Concentrations of analytes will be calculated according to the equations in the analytical methods cited. Outliers will be identified as those falling outside of the predetermined DQIG limits. Because of the nature of these studies, it is unlikely that any data will be omitted. Any outliers will be characterized to the fullest extent possible and identified as such. Whenever possible, analyses revealing outliers will be repeated. All data will be compiled and reported by the ARCADIS Work Assignment Leader.

5.2 Data Reduction

For the CEM, the data acquisition system logs all analyzed data and the computer software allows for various forms of data reduction. For the manual methods, the collected data are hand-input into Excel spreadsheets designed to reduce the data. CEM data will be plotted as pollutant concentration vs. time. Particulate and metals data will be presented in terms of concentration and emission rate (lb/hr).

5.3 Data Validation

After completion of the manual-methods hand-generated data in spreadsheets will be double-checked by the Sampling Engineer against the raw field data sheets for accuracy and completeness. CEM data will be reviewed for abnormalities or anomalies, prior to reporting. Laboratory reports will be reviewed to ensure that the reported data are qualified by the appropriate QA/QC information.

5.4 Data Reporting

Project progress will be reported to EPA in monthly progress reports and a final data report. The monthly progress report will be financial. Sampling data will be reported in the form of spreadsheets showing gaseous pollutant concentrations vs time and particulate and metals results in terms of concentration and emission rate (lb/hr).

6.0 REFERENCES

The following EPA Methods will be used as references for these tests. These methods are included as Appendix A to this QAPP.

- EPA Method 3A-*Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources*
- EPA Method 5-*Determination of Particulate Emissions from Stationary Sources*
- EPA Method 7E-*Determination of Nitrogen Oxides Emissions from Stationary Sources*
- EPA Method 10-*Determination of Carbon Monoxide Emissions from Stationary Sources*
- EPA Method 29-*Determination of Metal Emissions from Stationary Sources*

APPENDIX A

STANDARD METHODS

EPA Methods may be found on the internet at: <http://www.epa.gov/ttn/emc/promgate.html>

[Publications HOME](#)