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Final Technical Report

Development and Testing of the Advanced CHP System Utilizing the Off-Gas from the Innovative Green Coke Calcining Process in Fluidized Bed

Prepared by:

Gas Technology Institute

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End-Use Solutions
November 2013

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FINAL TECHNICAL REPORT
(August 1, 2009 to August 15, 2013)

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Table of Contents

APPENDICES:

EXECUTIVE SUMMARY

Green petroleum coke (GPC) is an oil refining byproduct that can be used directly as a solid fuel or as a feedstock for the production of calcined petroleum coke. GPC contains a high amount of volatiles and sulfur. During the calcination process, the GPC is heated to remove the volatiles and sulfur to produce purified calcined coke, which is used in the production of graphite, electrodes, metal carburizers, and other carbon products. Currently, more than 80% of calcined coke is produced in rotary kilns or rotary hearth furnaces. These technologies provide partial heat utilization of the calcined coke to increase efficiency of the calcination process, but they also share some operating disadvantages. However, coke calcination in an electrothermal fluidized bed (EFB) opens up a number of potential benefits for the production enhancement, while reducing the capital and operating costs.

The increased usage of heavy crude oil in recent years has resulted in higher sulfur content in green coke produced by oil refinery process, which requires a significant increase in the calcinations temperature and in residence time. The calorific value of the process off-gas is quite substantial and can be effectively utilized as an "opportunity fuel" for combined heat and power (CHP) production to complement the energy demand. Heat recovered from the product cooling can also contribute to the overall economics of the calcination process. Preliminary estimates indicated the decrease in energy consumption by 35-50% as well as a proportional decrease in greenhouse gas emissions. As such, the efficiency improvement of the coke calcinations systems is attracting close attention of the researchers and engineers throughout the world.

The developed technology is intended to accomplish the following objectives:

- Reduce the energy and carbon intensity of the calcined coke production process.
- Increase utilization of opportunity fuels such as industrial waste off-gas from the novel petroleum coke calcination process.
- Increase the opportunity of heat (chemical and physical) utilization from process off-gases and solid product.
- Develop a design of advanced CHP system utilizing off-gases as an "opportunity fuel" for petroleum coke calcinations and sensible heat of calcined coke.

A successful accomplishment of the aforementioned objectives will contribute toward the following U.S. DOE programmatic goals:

- Drive a 25% reduction in U. S. industrial energy intensity by 2017 in support of EPAct 2005;
- Contribute to an 18% reduction in U.S. carbon intensity by 2012 as established by the Administration's "National Goal to Reduce Emissions Intensity."

The ultimate goal of this effort is to reduce the energy and carbon intensity of the calcined coke production process. This goal is realized through the increased utilization of solid product heat and opportunity fuels such as process waste off-gas. The project designed and demonstrated an advanced CHP system concept to best utilize the off-gases as an "opportunity fuel." The off-gases contain a calorific and sensible heat of 1180-3600 Btu/lb of GPC, while the calcined coke contains a sensible heat of 725–900 Btu/lb of GPC. The total amount of recycled heat from the new developed coke calcination process has sufficient heat for the CHP to produce process stream and generate most of the electricity used by the fluidized bed. In order to successfully accomplish the project objectives a project team employed the integrated research and development (R&D) approach comprise of the Computational Fluid Dynamics **(**CFD) modeling, state-of-the-art information assessment, and bench-scale/pilot-scale evaluation.

The major benefits of the proposed technology are:

- Fuel savings due to use the opportunity fuel (off-gas from the industrial process).
- Significant energy savings due to integrated waste heat recovery (exhaust and bulk product).
- Lowering environmental impact due to improved combustion and reduced fuel consumption.

The final product of the undertaken effort is an advanced CHP system utilizing the integrated by-product/waste heat (off-gas, bulk heat) from the innovative energy-efficient process of petroleum coke calcination in fluidized bed. The above product is going to populate the growing industrial energy-intensive market of calcined coke production. The concept approach is developed and validated during the project. It is also expected to be successfully applied to the cross cutting markets such as metallurgical (steel production), power generation, and others.

CHAPTER 1

BACKGROUND AND PROBLEM STATEMENT

Green petroleum coke (GPC) is an oil refining byproduct that can be used directly as a solid fuel or as a feedstock for the production of calcined petroleum coke. GPC contains a high amount of volatiles and sulfur. During the calcination, GPC is heated in the calcining furnace to remove the volatiles and sulfur and to produce purified calcined coke, which is used in the production of graphite, electrodes, metal carburizers, and other carbon products. Currently, more than 80% of calcined coke is produced in rotary kilns or rotary hearth furnaces. These technologies provide partial heat utilization of the calcined coke to increase efficiency of the calcination process, but they also share some operating disadvantages. However, coke calcination in an electrothermal fluidized bed (EFB) opens up a number of potential benefits for the production enhancement, while reducing the capital and operating costs.

The increased usage of heavy crude oil in recent years has resulted in higher sulfur content in green coke produced by oil refinery process, which requires a significant increase in the calcinations temperature and in residence time. The calorific value of the process off-gas is quite substantial and can be effectively utilized as an “opportunity fuel” for CHP production to complement the energy demand. Heat recovered from the product cooling can also contribute to the overall economics of the calcination process. Preliminary estimates indicated the decrease in energy consumption by 35-50% as well as a proportional decrease in greenhouse gas emissions. As such, the efficiency improvement of the coke calcinations systems is attracting close attention of the researchers and engineers throughout the world. Coke calcination is a process that involves the heating of GPC in order to remove volatile material and purify the coke for further processing. Calcined coke is vital to the aluminum industry, where it is used to produce carbon anodes for aluminum production. The calcined coke is also widely used as recarburizer in iron and steel industry.

In recent years use of heavy, rather than the light crude oil for refinery process has been escalated. The resulting green coke produced from heavy crude oil processing has a higher sulfur content, which requires a significant increase in the calcination temperature and residence time. However, it is challenging to realize such a high temperature calcination process in conventional rotary kilns due to their complicated design, high heat losses, and increased capital and operating costs. This project aims to address this challenge through the development of the advanced CHP system using the energy from off-gas and calcined coke to produce process steam and generate the electricity used by the EFB process. An integrated system, using full recovery of by-product heat from off-gas and hot calcined coke product, will make the calcination process more economically feasible. This will be done through the reduction of energy consumption and a decrease in the capital and operating costs of the EFB furnace compared to conventional mechanical rotary or hearth furnaces.

CHAPTER 2

PROPOSED APPROACH FOR ADVANCED CHP SYSTEM

The technological approach being proposed here is an integration of the novel petroleum coke calcination process in a fluidized bed with an advanced CHP(ACHP) system utilizing the off-gases from EFB furnace and the heat from calcined coke cooling. The ACHP system produces two products: 1) low-pressure steam used in chemical process (for example, oil refining) and 2) high-pressure steam for electrical power generation for in-site use or selling to local grid. The amount of electricity generated from the ACHP system is about 90% of that needed by the fluidized bed, so the process is almost self-sustaining.

The approach is a combination of four technologies:

- Superior Graphite Company's (SGC) process of GPC calcination in EFB
- GTI's technology of off-gas combustion and cost-effective heat utilization
- Solex Thermal's technology of high temperature solid bulk product cooling
- Commercially available components for the CHP system (boiler, turbine, generator)

The proposed approach is illustrated in Figure 1. Chemical and thermal energy of the off-gas from fluidized bed calciner is utilized by combusting the off-gas in afterburner/boiler for electricity generation and process steam production. This ACHP system approach was chosen based on analysis and comparison different CHP schemes including GPC preheating, employing reciprocating internal combustion engine, etc. (Attachment B).

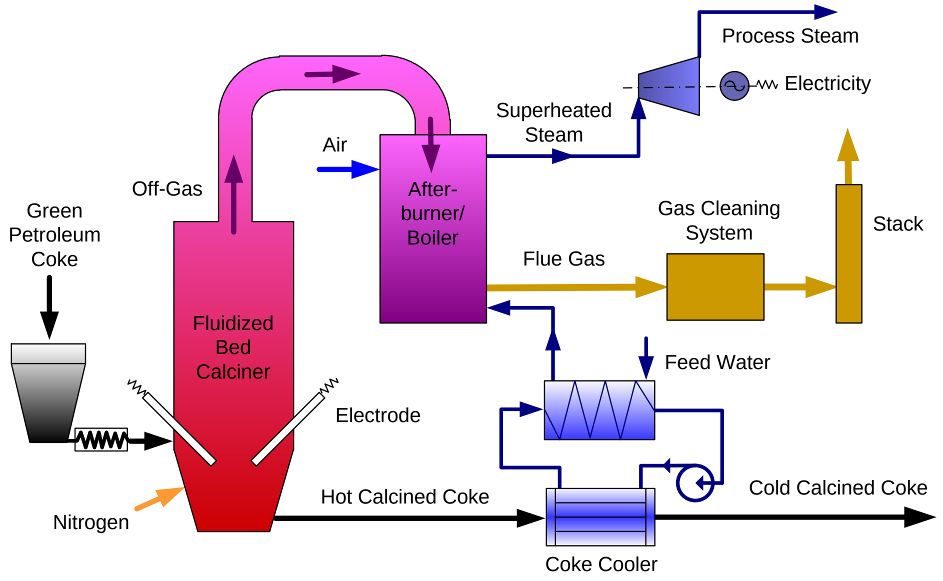


Figure 1. Combined heat and power generation system for advanced GPC calcination

Details for the off-gas combustion system is illustrated in Figure 2 below. The dimensions have been scaled down for a 6 ton/hr semi-commercial demonstration prototype.

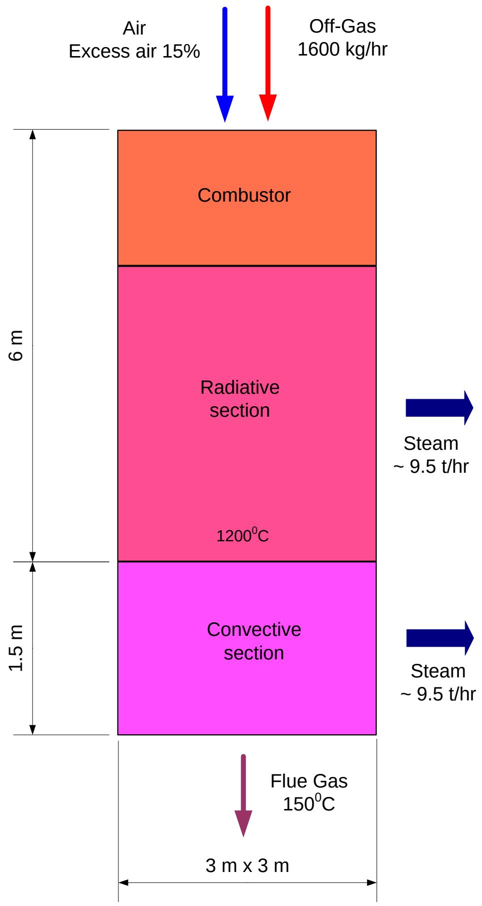


Figure 2. Schematic of the off-gas combustion system

Industrial process "opportunity fuels" have the advantage of being generated on-site, and are therefore a reliable source of "free" energy to be recycled back into the process or sold out to grid. The off-gases contain a calorific and sensible heat of 650–2000 kcal/kg of GPC (1180-3600 Btu/lb of GPC), while the calcined coke contains a sensible heat of 400–500 kcal/kg of GPC (725–900 Btu/lb of GPC). The total amount of recycled heat from the new developed coke calcination process has sufficient heat (1050–2500 kcal/kg of GPC or 1900–4500 Btu/lb of GPC) for efficient utilization in CHP system, depending on GPC composition, method of heat supply, required calcination performance, and process temperature.

EFB Process

SGC is developing an innovative process of petroleum coke calcination in fluidized bed with a production of calcined coke for anode manufacturing and regular applications such as metal carburizer, etc. Heat required for coke calcination is supplied by the complete combustion of the coke volatiles and partially of the carbon particles or from the direct transformation of electric power into the heat inside fluidized material. The off-gases and solid bulk product contain a large amount of heat (both chemical and sinsible) to be recycled and returned back into process (in either form of electric power or preheated raw material and fluidizing gas). Alternatively, such an exhausted energy can be sold as low pressure process steam for process heating applications or as electric power to the local grid. The waste heat utilization significantly improves the process efficiency, decreasing the amount of energy required for process conducting and minimizing the industrial emissions of carbon dioxide in the ambient environment.

Off-Gas Utilization

The off-gases from the EFB process contain a large amount of energy — calorific heating value and sensible heat due to its high temperature (650–2000 kcal/kg of GPC or 1180-3600 Btu/lb of GPC). Calculations of the adiabatic flame temperature of the off-gas indicated the order of 2100°C. This provides a strong potential for substantial release of the off-gases energy into a waste heat boiler, along with efficient burnout of the carbon particles that are carried out by this stream. GTI’s expertise in advanced combustion processes as well as decades of practical experience in gas-fired industrial equipment assured the project team with successful approach for the high-temperature off-gas combustion while producing low emissions of CO and NOx.

Bulk Product Cooling Heat Recovery

The calcined coke also contains a large amount of energy due to its high temperature. The sensible heat is 400–500 kcal/kg of GPC (725–900 Btu/lb of GPC). Solex Thermal's technology of heat utilization from bulk solid product cooling is incorporated to extract this energy and return it to the EFB process.

CHAPTER 3

FLUIDIZED BED FURNACE AND COKE CALCINATION PROCESS

The current work is based on:

- SGC experience with the development and commercialization of the new processes of high temperature heat treatment and production of carbonaceous materials in fluidized bed
- GTI experience in the area of various fuel effective combustion and waste heat utilization
- Solex Thermal Science Company in the area of solid grain product cooling builds upon past and current work in the technology area.

SGC developed a new concept of the following basic equipment for the developing a novel process of petroleum coke calcinations in fluidized bed:

- The concept of EFB furnace for coke calcinations with different method of heat supply;
- The concept of vertical cooler for calcined coke heat recycling;.

SGC performed a number of preliminary pilot-scale tests of developing process of petroleum coke calcinations in fluidized bed. It has demonstrated:

- The possibility of heat supply by volatile and partial carbon combustion or direct transformation of electric power in fluidized bed heat for coke thermal processing
- The possibility of calcined coke production with sulfur content ~ 2.5% at process temperature 1250–1300°C and ~ 1% at 1450–1475°C with bulk density 38–46 lbs/cf
- The possibility of full off-gas combustion in pilot water jacketed combustion chamber installed over gas flue by air adding.

CHAPTER 4

OFF-GAS COMPOSITION FROM FLUIDIZED BED FURNACE

Two GPC samples were taken for laboratory determination of proximate and ultimate analyses and heating value. The results are summarized in Table 1. Except for the sulfur and ash contents, both feed stocks are essentially similar. The first sample has higher sulfur content, 5.34% versus 3.20% for the second sample, but its ash content is lower, 0.70% versus 1.06% (the values are on a dry basis). The moisture and volatile matter fractions add up to 12.18% for the first sample and 12.87% for the second one. Heating values for the two fuels are also similar.

A target composition of calcined coke after high temperature (1000°C-1500°C) calcination should be as 98.5 to 99% carbon, less than 0.35% ash, and approximately 1% sulfur by weight.

Table 1. Proximate and Ultimate Analyses and Heating Value of GPC Samples
(Note: values in brackets are normalized)

GTI CRS Sample Login No. **Sample Description**	091544-001 Green Petcoke #1 (Shot Coke)-5.95% S	091544-002 Green Petcoke #2 (Sponge Coke)-3.7% S
Proximate Analysis (As received) (ASTM D5142, D5016)		
Moisture (107ºC), %	0.66	0.66
Volatile Matter, %	11.52	12.21
Ash (950ºC), %	0.71	1.06
Fixed Carbon, % (by difference)	87.11	86.07
Ultimate Analysis (Dry Basis) (ASTM D5373, D5016, D4239)		
Ash (950ºC), %	0.71 (0.70)	1.07 (1.06)
Carbon, %	90.19 (89.00)	91.50 (90.37)
Hydrogen, %	3.48 (3.43)	3.78 (3.73)
Nitrogen, %	1.55 (1.53)	1.66 (1.64)
Sulfur, %	5.41 (5.34)	3.24 (3.20)
Heating Value, BTU/lb (Dry Basis) (ASTM D5865)	15,260	15,550

Direct sampling of the product gas has not been possible at the CHP system design phase because of dilution at every location in the SGC furnace outlet that can safely be reached by a sampling probe. The GTI's Gasification Test Operations Support Group is providing assistance to the project team to develop a reasonable estimate of the off-gas composition for systems development and analysis work planned in the project.

Based on the information provided and available equipment capabilities, a combination of experimental and analytical work has been completed. We have analyzed the two GPC samples (proximate and ultimate analyses and heating value) and completed two TGA tests and one fixed-bed reactor test. The experimental data developed provided useful insights and guidelines for deriving a reasonable estimate for the off-gas composition.

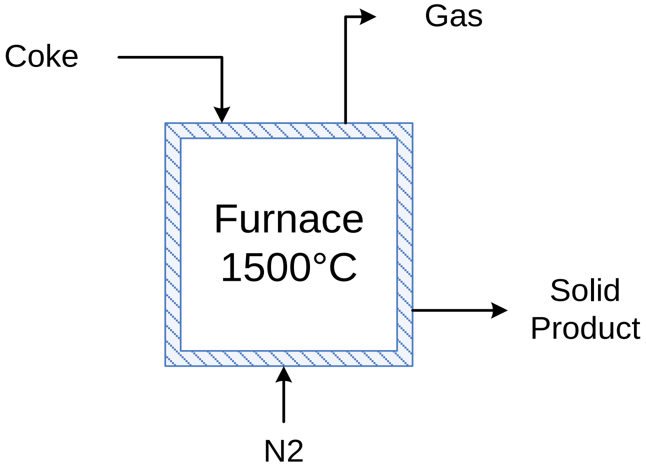


Figure 3. Schematic Diagram of the Petroleum Coke Calciner Furnace

This was accomplished using a commercial thermodynamic simulation software package, which was particularly useful in making projections into conditions not tested experimentally (i.e., 1050 to 1500°C temperature range) and insightful information about the sulfur species and other contaminants in the process gas. The recommended Calciner Furnace (Figure 3) off-gas composition is given in Table 2 below.

Table 2. Recommended Off-Gas Composition

H_2, vol%	72.99	CS_2, vol%	0.91	C_2H_2, ppmv	111
N_2, vol%	18.29	S_2, vol%	0.20	COS, ppmv	61.7
H_2S, vol%	4.04	HCN, vol%	0.20	NH_3, ppmv	8.7
CO, vol%	3.31	CH_4, vol%	0.05		

As we can see Ffrom the gas composition, the major components of the off-gas are hydrogen and nitrogen. The main source of hydrogen is volatiles from the GPC, and the main source of nitrogen is gas that is used for fluidization.

More details are provided in Appendix A.

Off-Gas Flow, Composition, and Combustion for different volatiles content

I. 5% volatiles (Dry GPC)
GPC load – 1400 kg/hr (dry)
Off Gas Inlet Temperature – 1500°C
Off Gas Flow – 140.18 kg/hr
Off Gas Composition, % volume
$H_2O = 0$
$CO_2 = 0$
$H_2 = 47.05$
$CO = 3.31$
$CH_4 = 0.13$
$N_2 = 38.67$
$H_2S = 10.84$
Off Gas Solids (carbon) – 68.84 kg/hr
Off Gas Solids (ash) – 0.22 kg/hr
Combustion Air Flow – 1418.3 kg/hr (1101.6 nm^3/hr)
Flue Gas Flow – 1627.54 kg/hr (1234.6 nm^3/hr)
Flue Gas Composition, % volume:
$CO_2 = 10.94$
$H_2O = 9.01$
$O_2 = 1.87$
$N_2 = 76.5$
$SO_x = 1.68$

II. 11.68% volatiles (Dry GPC)
GPC load – 1400 kg/hr (dry)
Off Gas Inlet Temperature – 1500°C
Off Gas Flow – 157.41 kg/hr
Off Gas Composition, % volume:
$H_2O = 0$
$CO_2 = 0$
$H_2 = 69.58$
$CO = 1.79$
$CH_4 = 0.09$
$N_2 = 22.65$
$H_2S = 5.89$
Off Gas Solids (carbon) – 86.98 kg/hr

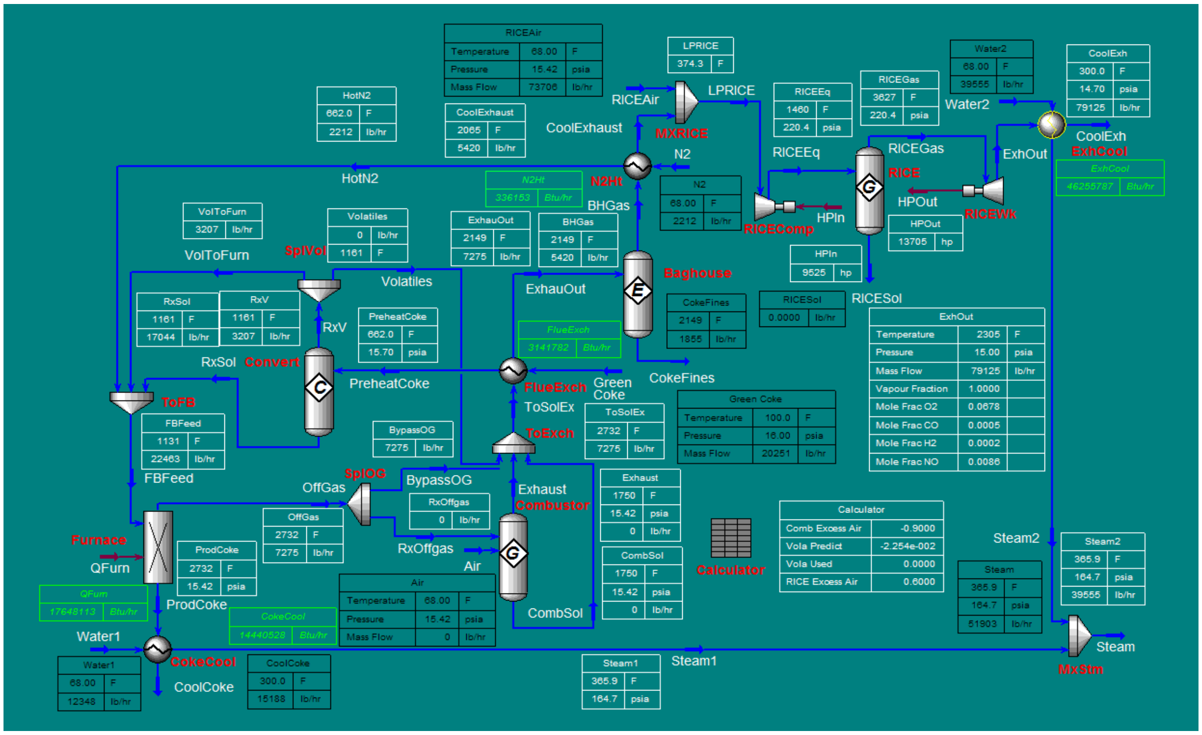
Off Gas Solids (ash) – 0.2 kg/hr
Combustion Air Flow – 2151.1 kg/hr (1670.9 nm^3/hr)
Flue Gas Flow – 2395.69 kg/hr (1882 nm^3/hr)
Flue Gas Composition, % volume:
  CO_2 = 8.97
  H_2O = 13.81
  O_2 = 1.86
  N_2 = 74.29
  SO_x = 1.08

III. 15% volatiles (Dry GPC)
GPC load – 1400 kg/hr (dry)
Off Gas Inlet Temperature – 1500°C
Off Gas Flow – 165.97 kg/hr
Off Gas Composition, % volume:
  H_2O = 0
  CO_2 = 0
  H_2 = 74.69
  CO = 1.44
  CH_4 = 0.08
  N_2 = 19.02
  H_2S = 4.77
Off Gas Solids (carbon) – 96.02 kg/hr
Off Gas Solids (ash) – 0.19 kg/hr
Combustion Air Flow – 2515.7 kg/hr (1954.1 nm^3/hr)
Flue Gas Flow – 2777.88 kg/hr (2204.06 nm^3/hr)
Flue Gas Composition, % volume:
  CO_2 = 8.42
  H_2O = 15.14
  O_2 = 1.86
  N_2 = 73.67
  SO_x = 0.91

CHAPTER 5

DEVELOPMENT OF THE CHP SYSTEM FOR GPC CALCINATION PROCESS

Critical parameters of the petroleum coke calcination process have been defined. Compositions of the waste off-gas from petroleum coke calciner at various production rates have been estimated experimentally and theoretically. The off-gas composition is important parameter for the CHP system development, since the off-gas is used as a fuel to generate heat and power from combustion. It was found that the off-gas contains essential amount of carbon particles. Some of the particles are entrained from the GPC. Other particles are formed as a result of pyrolysis of hydrocarbons from volatile matter of the GPC. The particle size distribution and off-gas composition have been estimated and used in combustion calculations. Two different approaches of the ACHP system to recover heat and power from coke calcinations process were considered using Aspen HYSYS 7.1 commercial software. The first one assumed the cooling the off-gas of interest to the safe temperature level and cleaned up to the conditions permitting the use it in power generation plant (turbine, engine). The second scheme assumed the immediate combustion of the off-gas flow that is coming out of the coke calciner with subsequent steam generation for power production and process needs. Figure 4 illustrates the potential schemes in Aspen HYSYS interface view. Based on extensive evaluation of both schemes, it was decided to further proceed with scheme B.



RICEAir		
Temperature	68.00	F
Pressure	15.42	psia
Mass Flow	73706	lb/hr

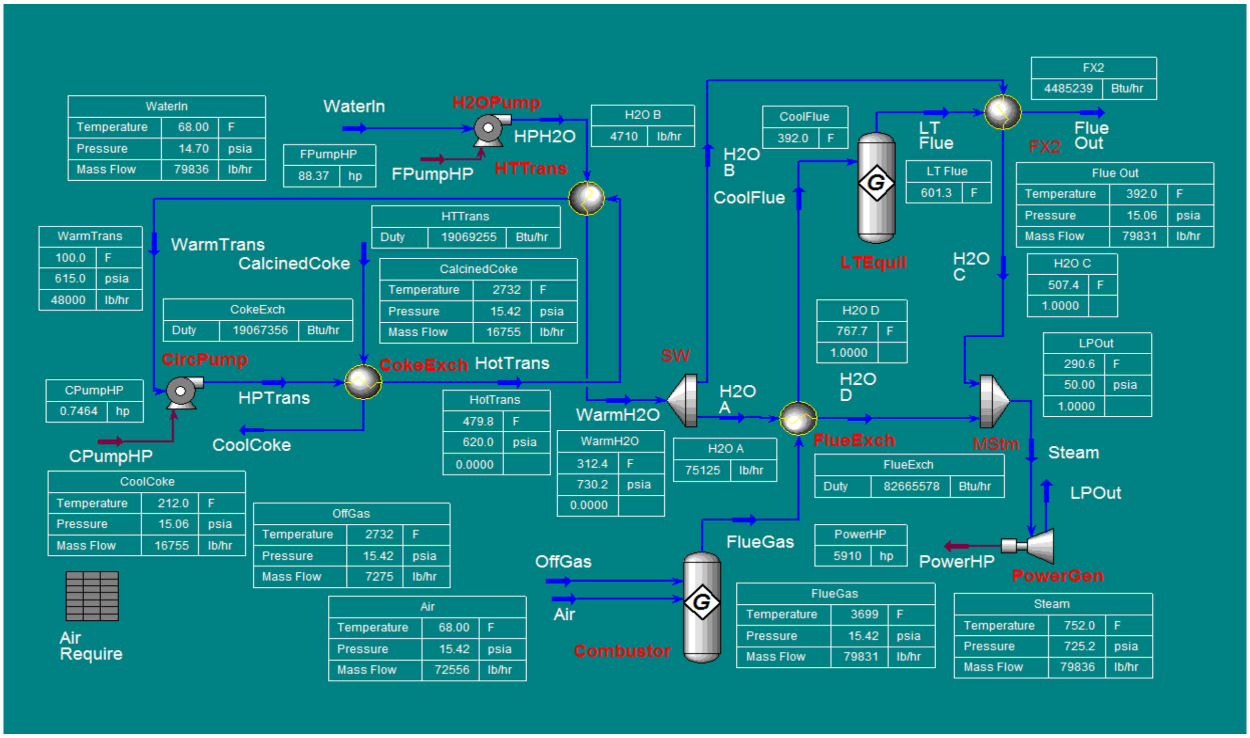
ExhOut		
Temperature	2305	F
Pressure	15.00	psia
Mass Flow	79125	lb/hr
Vapour Fraction	1.0000	
Mole Frac O2	0.0678	
Mole Frac CO	0.0005	
Mole Frac H2	0.0002	
Mole Frac NO	0.0086	

Green Coke		
Temperature	100.0	F
Pressure	16.00	psia
Mass Flow	20251	lb/hr

Calculator	
Comb Excess Air	-0.9000
Vola Predict	-2.254e-002
Vola Used	0.0000
RICE Excess Air	0.6000

Air		
Temperature	68.00	F
Pressure	15.42	psia
Mass Flow	0	lb/hr

A – CHP scheme with hot off-gas clean up for use in power generation plant



WaterIn		
Temperature	68.00	F
Pressure	14.70	psia
Mass Flow	79836	lb/hr

FPumpHP	
88.37	hp

H2O B	
4710	lb/hr

CoolFlue	
392.0	F

FX2	
4485239	Btu/hr

LT Flue	
601.3	F

Flue Out		
Temperature	392.0	F
Pressure	15.06	psia
Mass Flow	79831	lb/hr

WarmTrans	
100.0	F
615.0	psia
48000	lb/hr

HTTrans		
Duty	19069255	Btu/hr

CalcinedCoke		
Temperature	2732	F
Pressure	15.42	psia
Mass Flow	16755	lb/hr

CokeExch		
Duty	19067356	Btu/hr

H2O C	
507.4	F
1.0000	

H2O D	
767.7	F
1.0000	

LPOut	
290.6	F
50.00	psia
1.0000	

CPumpHP	
0.7464	hp

HotTrans	
479.8	F
620.0	psia
0.0000	

WarmH2O	
312.4	F
730.2	psia
0.0000	

H2O A	
75125	lb/hr

FlueExch		
Duty	82665578	Btu/hr

CoolCoke		
Temperature	212.0	F
Pressure	15.06	psia
Mass Flow	16755	lb/hr

OffGas		
Temperature	2732	F
Pressure	15.42	psia
Mass Flow	7275	lb/hr

PowerHP	
5910	hp

FlueGas		
Temperature	3699	F
Pressure	15.42	psia
Mass Flow	79831	lb/hr

Air		
Temperature	68.00	F
Pressure	15.42	psia
Mass Flow	72556	lb/hr

Steam		
Temperature	752.0	F
Pressure	725.2	psia
Mass Flow	79836	lb/hr

B – CHP scheme with complete off-gas combustion for power and heat production

Figure 4. Potential CHP scheme analysis with Aspen HYSYS 7.1

More details are provided in Appendix B.

CHAPTER 6

OFF-GAS COMBUSTION SYSTEM DEVELOPMENT, DESIGN, AND FABRICATION

Preliminary concept-design of the combustor for the advanced CHP system was developed by GTI. One of the critical issues to make the combustor to work efficiently is the sufficient residence time to burn out the carbon particles containing in the off-gas exhausting from the coke calcining furnace. GTI has assigned via subcontract S136 a specific combustion calculation procedure to the research team of University of Southern California (USC) with experience in similar analysis and is expected to provide GTI with the recommendations on combustion process optimization along with combustion efficiency estimate. Figure 5 illustrates the calculation domain under consideration by USC. Details are provided in Appendix C.

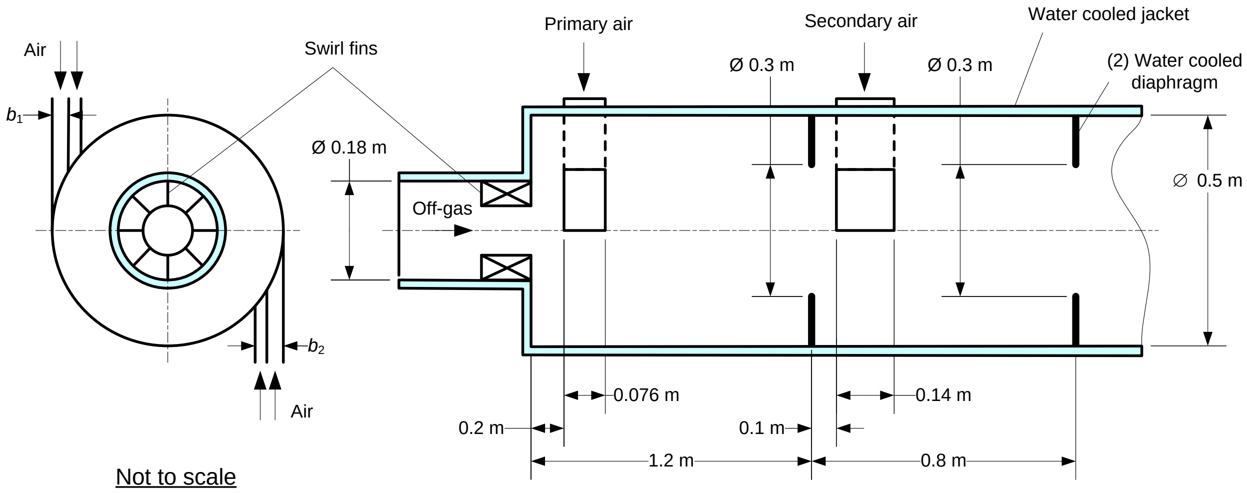


Figure 5. Calculation domain for combustion process optimization

GTI identified several potential candidates for ACHP design engineering and manufacturing (Figure 6). Alstom Power Energy Recovery (APER), formerly known as American Schack Co. (Wexford, PA) was selected to be the project partner as the combustor developer and supplier. Upon extensive technical consideration between project partners and Alstom technical staff GTI has subcontracted Alstom for engineering feasibility study of advanced CHP design (subcontract S175) followed by the equipment manufacturing and supply phase.

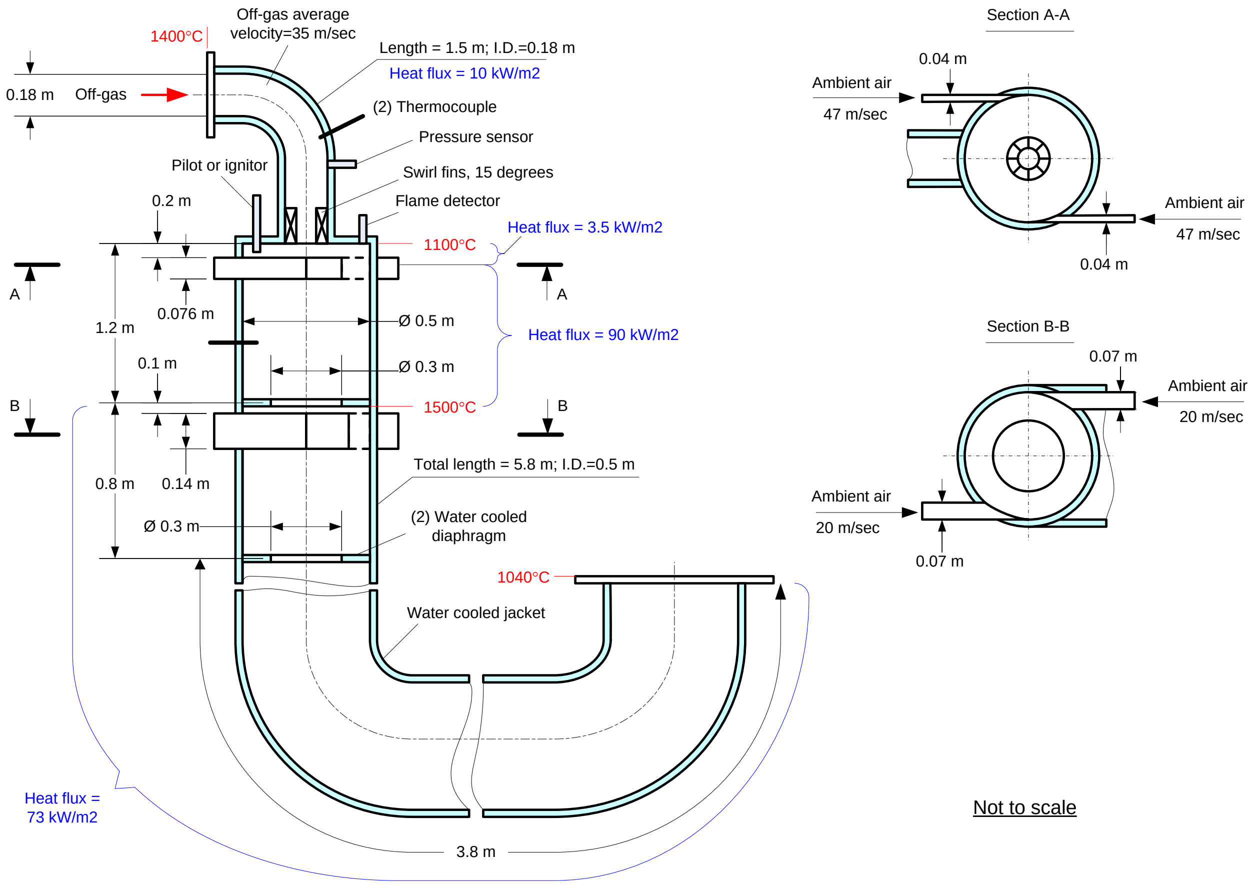


Figure 6. Preliminary CHP concept-design by GTI

Table 3. Combustion air nozzles dimensions

	Width, m	Height b_1, b_2, m
Primary air (2 nozzles)	0.076	0.04
Secondary air (2 nozzles)	0.14	0.07

Flow parameters:

- off-gas flow rate: 245 kg/hr
- excess air: 10%
- combustion air flow rate: 3123 kg/hr
- primary combustion air flow rate: 60% (33088 SCFH)
- secondary combustion air flow rate: 50% (27573 SCFH)
- secondary combustion air is oxygen enriched and contains 23% of oxygen
- flue gas flow rate at the combustion chamber outlet: 70691 SCFH
- combustion air temperature: 20°C
- swirling flow angle: 15 degrees

Off-gas composition (% mass): N_2 (nitrogen) – 39.76%; ash – 0.08%; carbon particles (C) – 35.56%; CH_4 (methane) – 0.09%; CO (carbon monoxide) – 3.14%, H_2 (hydrogen) – 9.53%; S_2 (sulfur) – 11.84%

Additional information:

- total surface area of combustion chamber (ID=0.5m) and radiant section (ID=0.5m): 13.5m^2
- total heat flow in the off-gas pipe (L=4 m, ID=0.18m): 83.5 kW
- total heat flow in combustion chamber and radiant section: 971 kW

CHP general arrangement design has been developed jointly by GTI (technology developer), SGC (host plant in Hopkinsville, KY) and APER (US leading engineering company with the strong experience in high temperature and special services industrial equipment). Figure 7 below illustrates the advanced CHP 3-D general arrangement. The vertical orientation has been selected to ensure minimal deposition on the advanced CHP wall in case of non-expected operation, if any. The parallel zoned water cooling along with intermediate water circulating loops is to optimize the high flow rate without starving the existing cooling water system.

The detailed design basis has been developed jointly by GTI, SGC, and APER through multiple communications, site visits, and data iterations. Taking into account the fact that off-gas composition and temperature level are very different as opposed to existing calciners, the ACHP combustion chamber required the special design and process calculation methods. Most of the technical input and calculation design methods are proprietary to SGC and APER and excluded from this report.

More details are provided in Appendices C and D.

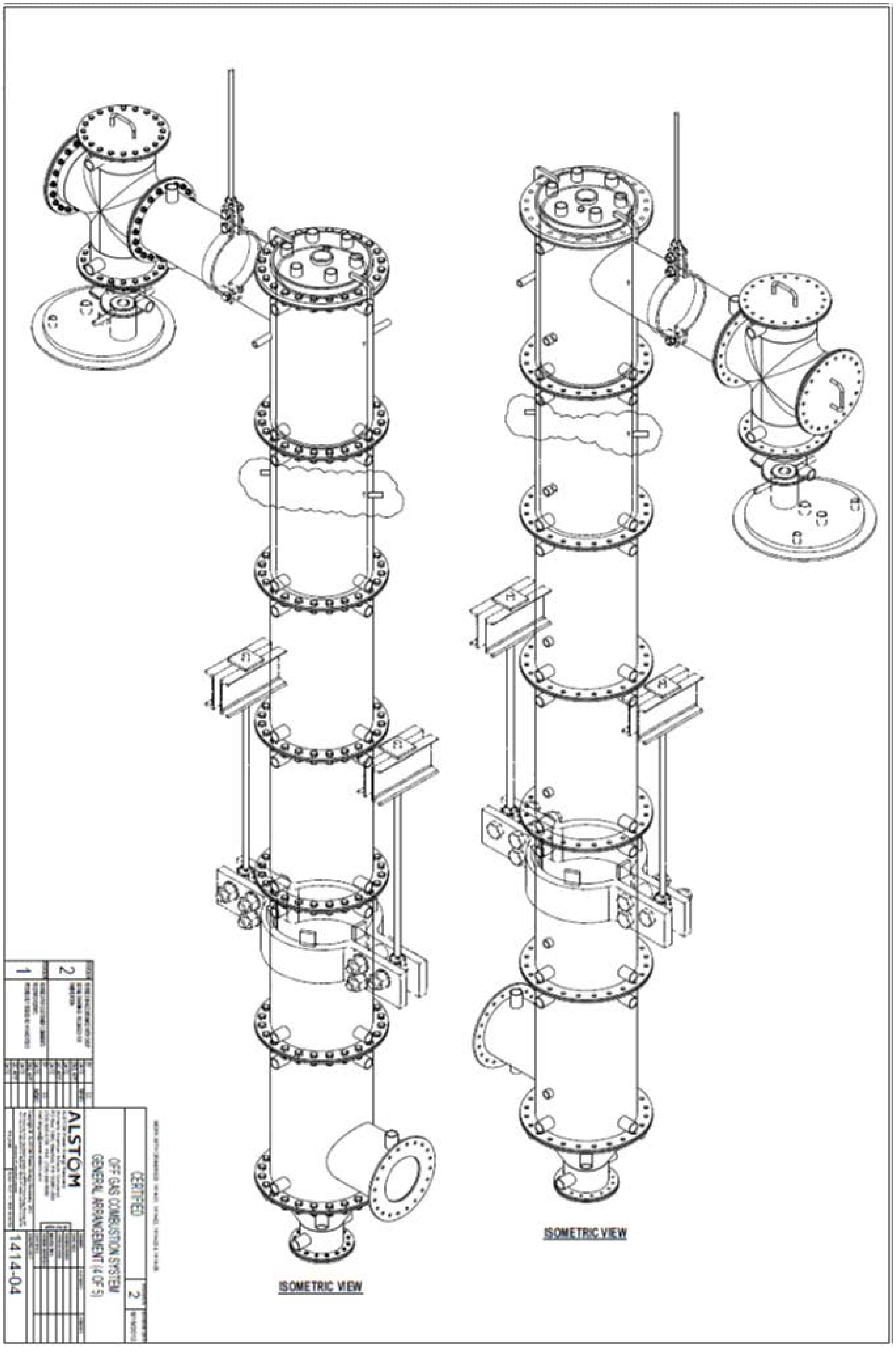


Figure 7. Advanced CHP General Arrangement [by courtesy of APER]

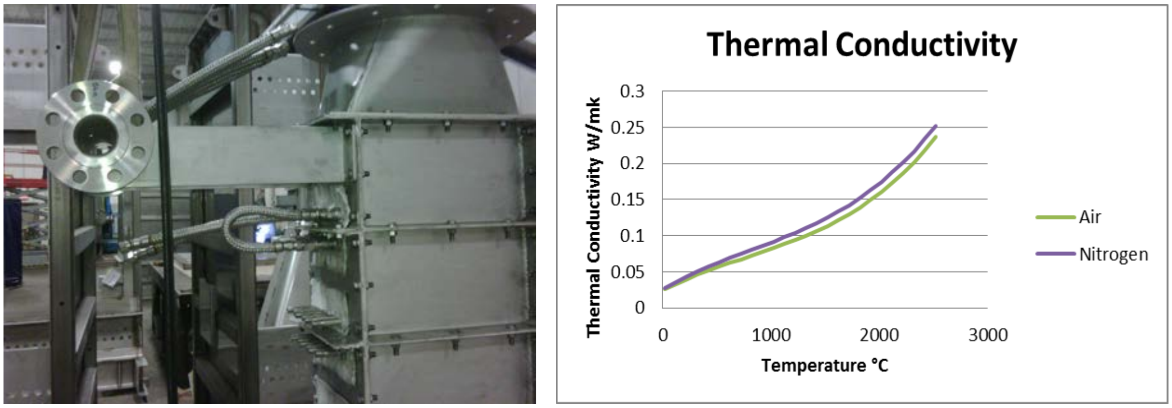
CHAPTER 7

SOLEX CALCINED COKE COOLER THERMAL DESIGN

Bulk solid product cooling approach has been developed by Solex Thermal Science under subcontract S124 and pilot-scale unit was fabricated and evaluated by the Solex team in laboratory as well as field environment. Prior to file test, the Solex team ran the original thermal models using an average thermal conductivity of the product measured at ambient conditions. Figure 3a illustrates the pilot-scale hot bulk solids cooler for the calcined coke product installed at Solex's test facility. Since the thermal conductivity of the air or nitrogen in the void space of the particles increases significantly with temperature, the thermal conductivity of the bulk solid also increases significantly with temperature, as show in the chart in Figure 3b.

The major observations during the testing were:

- cooling performance of the test unit was much better than expected;
- casing remains cool to the touch during testing except at very top of top bank;
- plates looked like new when testing was completed.



a b

Figure 3. Hot bulk solid cooler (a) and bulk product thermal conductivity versus carrier gas temperature (b)

More details are provided in Appendix E.

CHAPTER 8

OFF-GAS COMBUSTOR EXPERIMENTAL SETUP, MEASUREMENT SYSTEM, AND EQUIPMENT

As the first step of the ACHP system development, it was decided to refine a design of the off-gas combustion system and evaluate it in order to confirm that the off-gas can be efficiently combusted, including the combustion of carbon particles in the off-gas. Aspen HYSYS modeling results for ACHP system as well as estimated GPC and off-gas compositions were used to develop the off-gas combustion system.

The main component of the system is afterburner (combustion chamber), see Figure 8a. Also, the combustion system was tested without producing steam for power generation. In this case, the combustion chamber was designed with water cooled walls to simulate a boiler. The pilot off-gas combustion chamber was designed per SGC's specifications, fabricated by APER, field engineered and installed by SGC contractors and SGC research facility (Figure 8b).

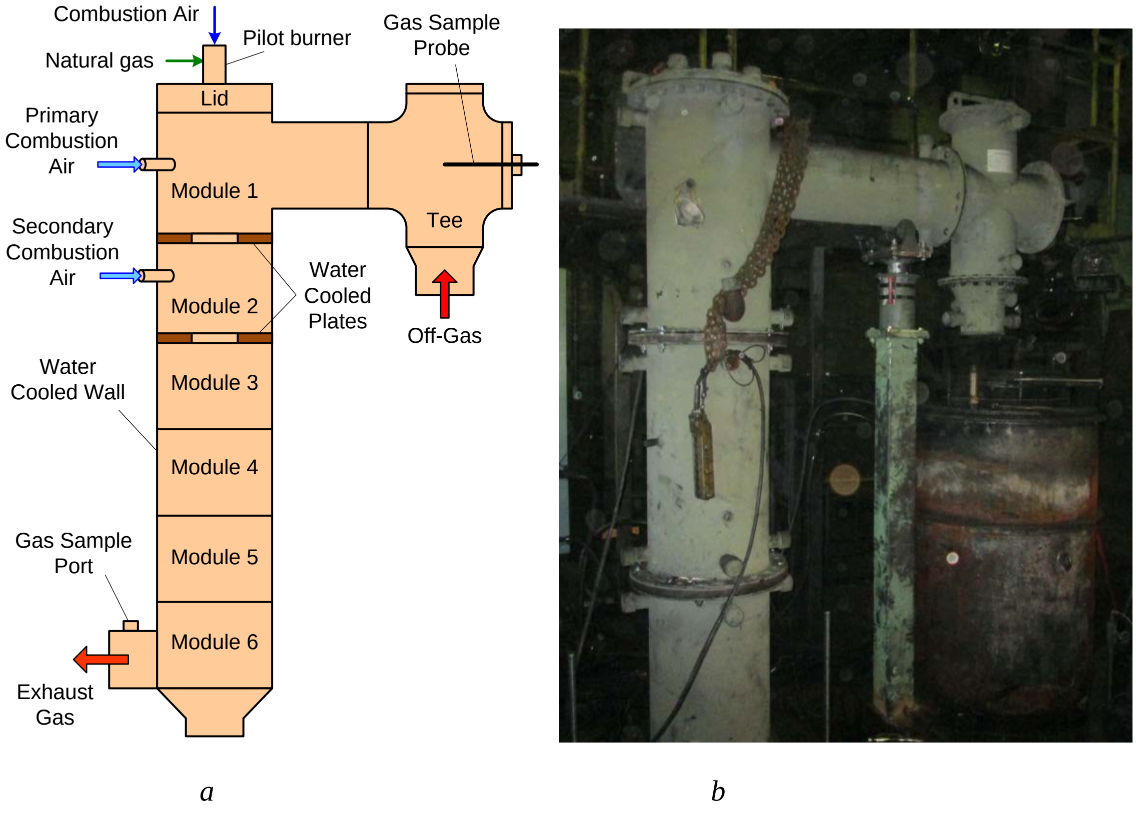


Figure 8. Afterburner configuration (*a*) and the top sections of installed system (*b*)

The combustion chamber consisted of a tee section connecting the EFB calciner with off gas combustion system, pilot (ignition) burner on the top of the chamber, six water cooled small modules to allow flexibility in the system layout and to minimize installation problems, combustion air tangential nozzles, and exhaust gas outlet (Figure 8a). The off-gas is supplied from the EFB calciner to the combustion chamber through a tangential hole in refractory liner. There are two primary combustion air slots (nozzles) in module 1 and two secondary combustion air slots (nozzles) in module 2. The nozzles are near radial to create swirl flow in the combustion chamber. It is expected the tangential flow of fuel and air in the primary and secondary sections will have a "cyclone separator" effect resulting in particle residence times much longer than the gas residence time, to allow gasification and complete combustion of particles. It is also expected that normally more than 50 % of the total theoretical air will be required for the primary stage.

Measurement System

The testing procedure included warm up the calciner first then introduce GPC for calcinations. When the volatiles from the calciner reached the afterburner, the controlled combustion process of the off-gas was taken place and heat was recovered. Parameters setting at GPC feed rate 22 lb/hr – 104 lb/hr; EFB calciner process temperature 1000°C – 1200°C; and combustion air flow rate adjusted to provide the optimal excess air for efficient combustion of the off-gas. Off-gas composition was measured by Varian micro gas chromatograph and exhaust gas composition was measured by Horiba gas analyzer during the experiment.

Primary and secondary combustion air flow rates were set to provide ~10% excess air in the cyclonic combustion chamber. The air flow rates were controlled by air flow meters installed at each air line. The startup procedure of the combustion system included ignition of the natural gas pilot burner first before the off-gas is introduced into the off-gas combustion system. A flame detector was installed on the pilot burner and indicated the gas pilot was in operation. There was a pilot flame in the off-gas combustion chamber. When the pilot flame was established, the off-gas was introduced in the combustion chamber. Thermocouple readings (temperature rise in the combustion chamber in the beginning of the process) were observed to be sure the off-gas was combusted. When the main flame in the off-gas combustion chamber was established and the system operated properly, negative gas pressure in the combustion chamber and excess air were controlled to keep nominal values of the parameters. The value of the gas pressure was dictated by the process and controlled by the exhaust gas fan.

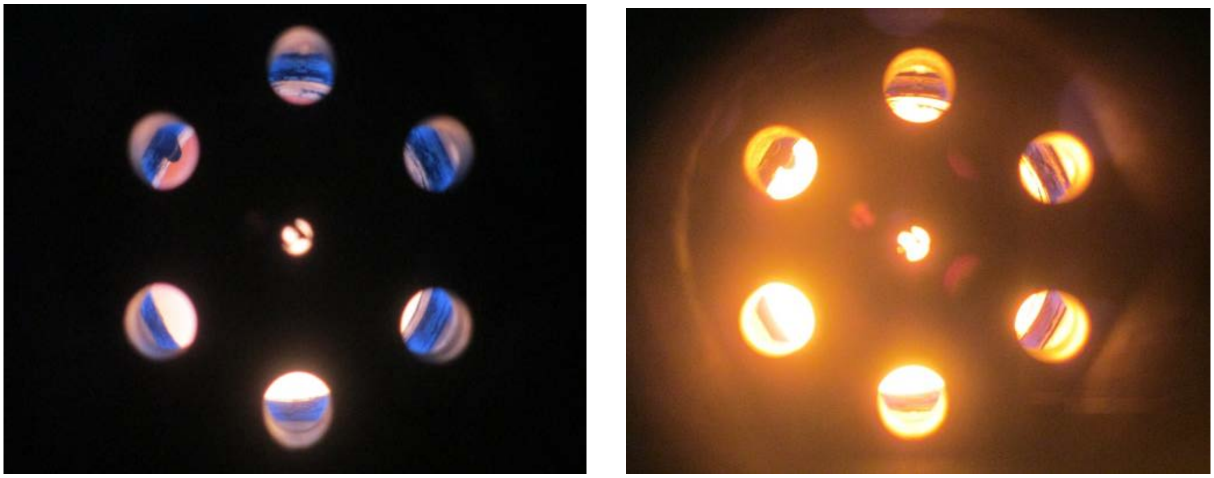
More details are provided in Appendices F and G.

CHAPTER 9

FLUIDIZED BED CALCINER AND OFF-GAS COMBUSTOR TESTING, RESULTS, AND DISCUSSIONS

GPC Calcination and Afterburner Test Results and Discussions

With adjustment of operating conditions and pilot burner control, a stable operation of the off-gas combustion system was established. The system was running stable at the required GPC feed rates from 22 lbs/hr to 104 lb/hr. Figure 9 shows the flame image in combustion chamber when the pilot burner is on (pos. *a*) and off (pos. *b*). The combustion of the off-gas with high content of hydrogen and carbon particles in the off-gas resulted in yellow flame. Visual observation through the view port of the pilot burner allowed concluding a stable cyclonic combustion in the combustion chamber. During the whole trial no rodding of the off-gas nozzles was needed. Carbon deposit was found in the afterburner combustion chamber gaps at lower GPC feed rate when the off-gas flow rate was also low and the "cyclone effect" in the afterburner was not sufficient to provide complete combustion of carbon particles.



a *b*

Figure 9. Off-gas combustion in afterburner (view from observation port): (*a*) pilot burner on, (*b*) pilot burner off

The volatile in the GPC was 10.1%, and dropped to 0.24% after calcination at 900°C. Ash content in the green coke dropped from 0.32% to 0.13%. Sulfur dropped from 5.90% to 1.64%. Figure 10 shows the off-gas composition versus GPC feed rate. The off-gas composition is in a good agreement with theoretical and experimental predictions described above.

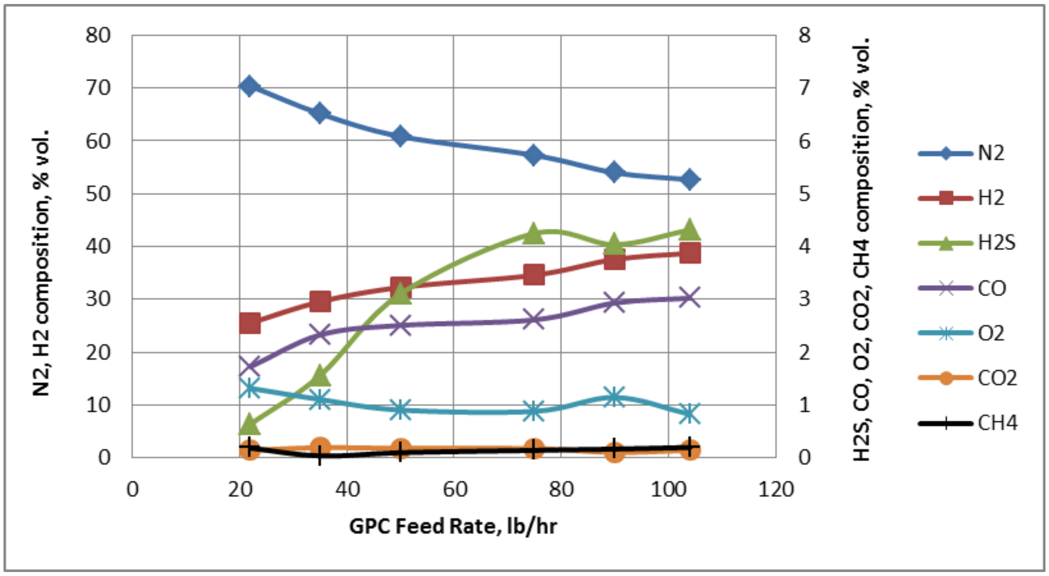


Figure 10. Off-gas composition versus GPC feed rate

Figure 11 shows a typical gas temperature distribution in afterburner symmetry axis. Based on the measured temperature and gas composition in the combustion chamber as well as measured cooling water temperature in the walls channels, the heat balance of the system was done. The cooling water temperature only increased about 1~1.5°C after passing through the cooling channels during the trial, which was also confirmed by the heat balance analysis.

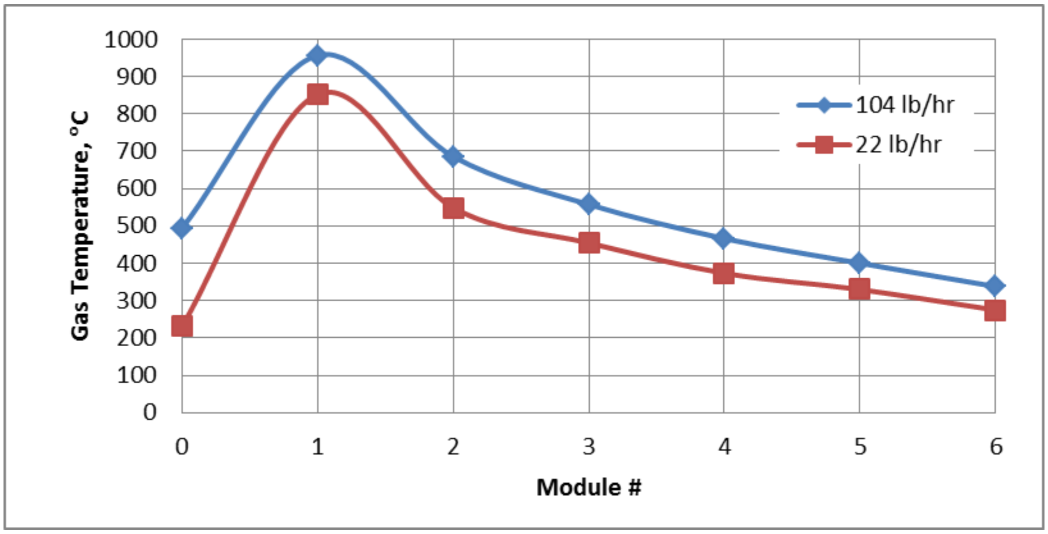


Figure 11. Typical gas temperature distribution in afterburner symmetry axis

Typical composition of afterburner exhaust gas was the following: N_2~88.7%, O_2=4%, CO_2=7.3%, NO_x=24 vppm, CO=33 vppm, SO_2>0.5%.

CHAPTER 10

CONCLUSIONS

- Advanced CHP system for petroleum coke calcinations in electrothermal fluidized bed was developed and theoretically evaluated.
- Off-gas composition from fluidized bed furnace has been experimentally and theoretically evaluated. This off-gas composition was used to design the off-gas combustion system.
- Calcined coke cooler was developed, designed, fabricated, and tested to demonstrate the feasibility of capturing and utilizing the thermal energy that would be available from bulk solid product cooling
- Afterburner (combustion chamber) for the advanced CHP system to combust off-gas from GPC calciner was developed, designed, fabricated, and tested. The combustion chamber consisted of a tee section connecting the fluidized bed furnace, pilot (ignition) burner, six water cooled modules, combustion air tangential nozzles, and exhaust gas outlet. The combustion air nozzles created swirl flow in the combustion chamber which acted as a "cyclone separator" for better combustion of coal particles.
- Successfully created stable controlled high-efficiency combustion in the afterburner for throughput from 22 lbs/hr to 104 lb/hr. System pressure and temperature stabilized with pilot burner always on, stable combustion was observed in the combustion chamber. First time ever proved the concept of controlled combustion of volatiles from GPC calcination.
- Proved the concept of volatile combustion for energy recovery.
- The green coke was well calcined during the low throughput test. No flue rodding was needed during the trial. Furnace pressure was maintained at slightly positive pressure.
- The volatile in the green coke is 10.10%, and dropped to 0.24% after calcination at 900°C. Ash content in the GPC dropped from 0.32% to 0.13%. Sulfur dropped from 5.90% to 1.64%.
- The conducted research and development efforts of the CHP system for GPC calcining process have confirmed feasibility for predicted energy savings of the proposed technology, namely, for a standard production unit of 100,000 Mt/yr, the energy savings would be 0.806 TBtu/yr (steam) or 0.940 TBtu/yr (natural gas). At that, the savings in energy intensity would be 9.4 MMBtu/Mt.

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APPENDIX A

Estimation of composition of off-gas from fluidized bed calciner

Fuel Characterization

The two petcoke samples, “Shot Coke” and “Sponge Coke” were submitted to GTI’s Environmental and Chemical Services Laboratory (ECRS) for determination of proximate and ultimate analyses and heating value. The results are summarized in Table 1. Except for the sulfur and ash contents, both feedstocks are essentially similar. The “Shot Coke” has higher sulfur content, 5.34% versus 3.20% for the “Sponge Coke”, but its ash content is lower, 0.70% versus 1.06% (all values are on a dry basis). The moisture and volatile matter fractions add up to 12.18% for the “Shot Coke” and “12.87% for the “Sponge Coke”. Heating values for the two fuels are also similar, as shown in Table 1.

No sample from the Calciner Furnace solid product material was available for chemical analysis at GTI, but typical specifications provided by Superior Graphite, indicate the treated material contains 98.5 to 99% carbon, less than 0.35% ash, and approximately 1% sulfur. It is not clear if these specifications apply to a particular petcoke or to both materials.

Table 1. Proximate and Ultimate Analyses and Heating Value of Green Petcoke Samples
[Note: values in ( ) are normalized]

GTI CRS Sample Login No. **Sample Description**	091544-001 Green Petcoke #1 (Shot Coke) - 5.95% S	091544-002 Green Petcoke #2 (Sponge Coke) - 3.7% S
Proximate Analysis (As received) (ASTM D5142, D5016)		
Moisture (107ºC), %	0.66	0.66
Volatile Matter, %	11.52	12.21
Ash (950ºC), %	0.71	1.06
Fixed Carbon, % (by difference)	87.11	86.07
Ultimate Analysis (Dry Basis) (ASTM D5373, D5016, D4239)		
Ash (950ºC), %	0.71 (0.70)	1.07 (1.06)
Carbon, %	90.19 (89.00)	91.50 (90.37)
Hydrogen, %	3.48 (3.43)	3.78 (3.73)
Nitrogen, %	1.55 (1.53)	1.66 (1.64)
Sulfur, %	5.41 (5.34)	3.24 (3.20)
Heating Value, BTU/lb (Dry Basis) (ASTM D5865)	15,260	15,550

Ash not corrected for SO_3 content.

Thermogravimetric Analysis (TGA)

Initially, one TGA test was conducted in late August 2009 using a 400.12-milligram sample of the “Shot Coke” material. The facility used, a Thermo Cahn TherMax 500 TGA unit, capable of operating at temperatures as high as 1100°C and pressures up to 70 bar, is shown in Figure 2. TGA tests measure the change in sample weight over time as a sweep gas flows over a small sample (usually 20 to 100 mg) in a small reactor tube. The unit is not equipped with any off-gas analysis instrument. This facility is not particularly suitable for conducting testing with carbonaceous feedstocks because of the limitation of having to have the sample in the reaction chamber as test conditions (temperature, pressure, and reaction gas mixture) are established. However, it was used in this case since the main purpose was to develop some technical insights into petcoke weight loss (rate and extent) at different temperature increments, up to a maxium temperature of approximately 1050°C (our other TGA unit is limited to about 950°C). The results, presented in Figure 3, appear to suggest the sample loses weight at essentially the same rate as temperature was raised incrementally and maintained at the temperature levels and time intervals indicated. Overall, the total weight loss was estimated at 13.1%, which is qualitatively consistent with the sum of the moisture and volatile matter fractions in the fresh material.

A second TGA test was conducted in early October 2009 with a similar sample size (399.95 mg) of the same petcoke material ("Shot Coke"). The main goals of this test were to: 1) establish results reproducibility, conduct testing at 1050°C for a more extended period permitting the sample weight to stabilize, promote further petcoke devolatilization by using a higher sweep gas (N_2) flow than in the first TGA test (1000 versus 50 cm^3/min), and provide a post-test sample for characterization to relate solids composition data from GTI's lab samples (treated at the highest temperature of 1050°C) to actual process samples (treated at 1500°C). Test results are presented in Figure 4.

A summary of test conditions and results is provided in Table 2 [Note: data reported in the fourth column relate to a different test that is discussed later in this report]. Despite slight differences in operating conditions (sweep gas flowrate, testing duration at a given temperature, etc.), test results are essentially reproducible, both suggesting the "Sponge Coke" sample lost a total weight corresponding to the sum of its moisture and volatile matter fractions, as can be expected. Furthermore, there is excellent agreement between the sample weight loss calculated based on TGA data and that based on measurements of sample weight made using a separate balance before and after testing on fresh and reacted samples, respectively.

Table 2. TGA and Devolatilization Test Results

	TGA Test No. 1	TGA Test No. 2	CTF Test No. 1
Date	8/25/2009	10/2/2009	8/27/2009
Initial Sample Mass	400.12 mg	399.95 mg	50 g
N_2 Sweep Rate	0.05 slpm	1 slpm	0.6 slpm
Test Duration	385 min.	500 min.	297 min.
% mass lost (TGA)	13.102% (52.4 mg)	10.072% (40.279 mg)	---
% mass lost (Lab Balance)	12.94% (51.8 mg)	10.25% (41.01 mg)	10.66% (5.33 g)

The post-test sample from the second TGA test, weighing approximately 358.94 mg, was submitted to the ECRS Lab for ultimate analysis determination. Because of the limited sample size, ECRS could only determine C, H, N, and S; there was not sufficient sample to run an ash analysis and get any meaningful weight of "residue". The results are summarized in Table 3. The C/H/N/S data, reported by ECRS strictly as-analyzed and on a moisture-free basis, were first normalized and then minor corrections were made to account for the presence of about 2.8 mg ash (0.78%) in the post-test sample. Table 4 also calculates the overall weight loss (i.e., extent of reaction) for the different ultimate analysis components (moisture, ash, C, H, N, and S). As shown, while approximately 96% of the H component reacted, only about 6% of the C did. The N reaction extent was estimated at about 30.4% and that for S was limited to about 9%.

Table 3. Post-test Sample Ultimate Analysis and Conversion Estimates for the Various Components (ECRS Login No. 091578-001)

	Hi-S Petcoke					
Ultimate Analysis (As Received)	Ult. Anal., Fresh	mg, TGA Test #2 Feed	Ult. Anal., Reacted (normalized)	Ult. Anal., Reacted (corrected for ash content)	mg, TGA Test #2 Reacted	Wt. % Loss
Moisture (107°C), %	0.66	2.6	0.09	0.09	0.3	87.86
Ash (950°C), %	0.70	2.8		0.78	2.8	0.00
Carbon, %	88.41	353.6	93.15	92.43	331.8	6.18
Hydrogen, %	3.41	13.6	0.16	0.16	0.6	95.90
Nitrogen, %	1.52	6.1	1.19	1.18	4.2	30.40
Sulfur, %	5.30	21.2	5.41	5.37	19.3	9.09
	100.00	399.95	100.00	100.00	358.94	10.25

These results suggest that an appropriate flow reactor test at 1050°C can yield reliable <u>overall</u> product gas composition data, i.e., H_2, H_2O, CO, CO_2, CH_4, N_2. However, only testing at much higher temperatures can provide contaminants concentration data, such as sulfur speciation, since only about 9% of the sulfur reacted at 1050°C. Nevertheless, by making reasonable assumptions the data developed based on fuel characterization and TGA testing were used to make projections into conditions not tested experimentally, as explained below.

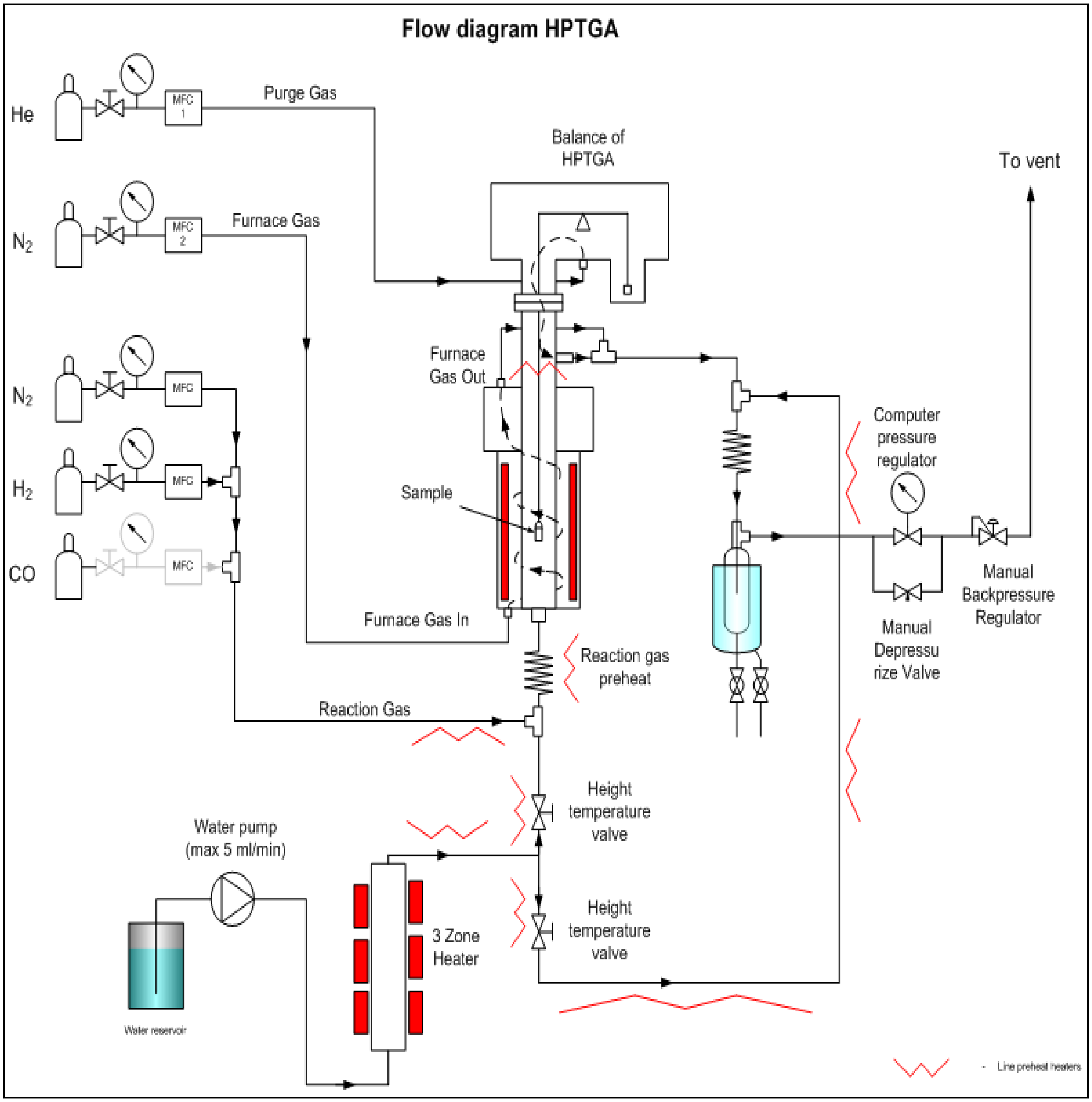

Figure 1. Schematic Diagram of GTI’s High-Pressure Thermogravimetric Analyzer

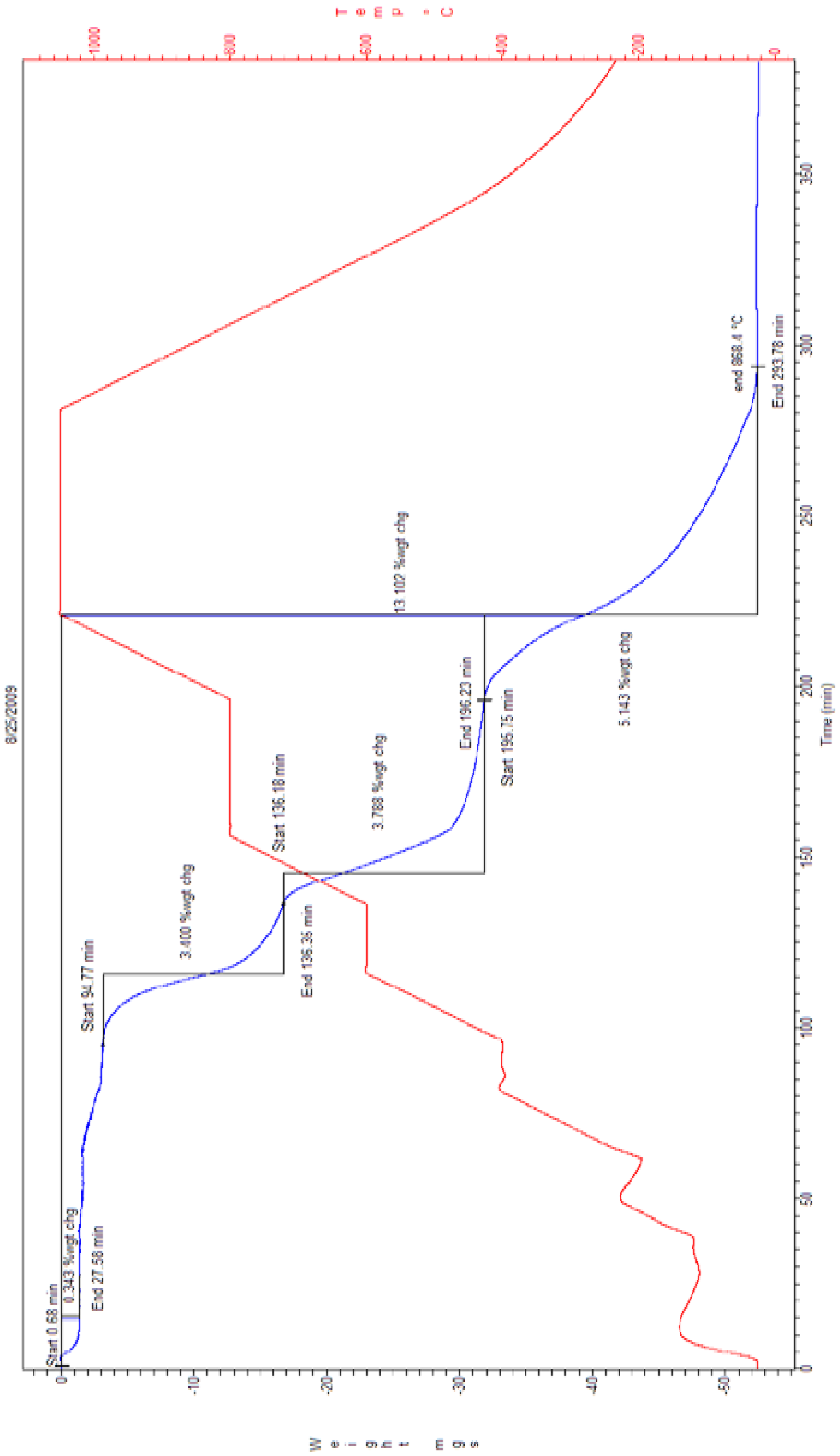

Figure 2. Test No. 1 (400.12 mg initial sample; 0.05 slpm N_2 sweep gas; 13.1% wt loss)

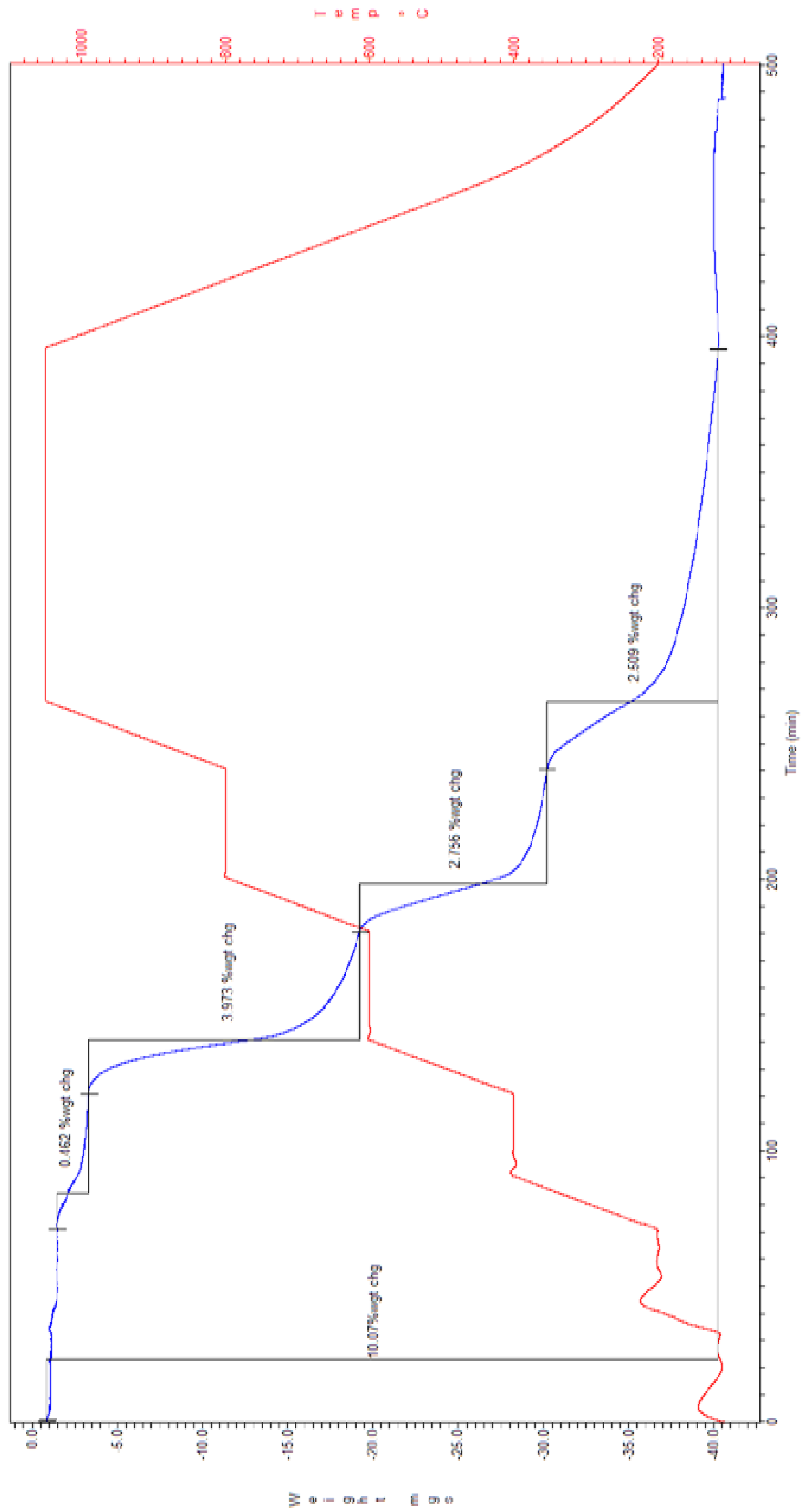

Figure 3. TGA Test No. 2 (399.95 mg initial sample; 1 slpm N_2 sweep gas; 10.07% wt loss)

HSC Thermodynamic Simulation

The analytical and TGA test results were used to conduct preliminary thermodynamic simulations to estimate the equilibrium composition of the Calciner Furnace exhaust gas. The HSC software package (HSC Chemistry 6, v. 6.12, Outotec Research Oy, Antti Roine, www.outotec.com) was used. Among other things, this thermodynamic simulation software can estimate the equilibrium amounts and concentrations of several phases and numerous chemical species that are specified by the user based on the Calciner Furnace feed. This latter consists of petroleum coke ("Shot Coke" or "Sponge Coke") and nitrogen (100 kg N_2/tonne of petcoke). Inputs to the HSC program are specified in Table 4 for both types of petcokes based on their ultimate analyses and a number of assumptions that area explained in Table 5 along with their implications to how species are inputted. The moisture, H, and N components were assumed to undergo 100% conversion at 1500°C, which is a reasonable assumption based on TGA test results at the lower temperature of 1050°C. The Calciner Furnace solid product is assumed to contain 1% S based on the specifications provided by Superior Graphite. A major assumption had to be made for carbon conversion. TGA results showed approximately 6% of the carbon was converted at 1050°C; 10% conversion is assumed in the HSC thermodynamic simulation. Based on this assumption, the overall sulfur conversion was estimated at approximately 84.9%. Because the HSC program does not accept "petroleum coke" as an input species, the solid feed to the Calciner Furnace was inputted as solid carbon. For the converted elements of the feed material, H was introduced as H_2, N as N_2, S as S_2, and reacted C as 50/50 CO/CO_2. For the product gas, the list of possible gaseous species considered included: N_2(g), CH_4(g), C_2H_2(g), C_2H_4(g), C_2H_6(g), CO(g), CO_2(g), COS(g), CS_2(g), H_2(g), HCN(g), H_2O(g), H_2S(g), N_2(g), NH_3(g), NO(g), NO_2(g), NO_3(g), O_2(g), S(g), S_2(g), S_8(g), SO_2(g), SO_3(g).

Thermodynamic simulation results for the higher sulfur case ("Shot Coke") are presented in Table 5 and graphically in Figures 5 through 7. Table 5 provides the equilibrium concentrations of all species considered. Figure 5 shows the equilibrium amounts of major species including the solid carbon phase. Figure 6 shows the equilibrium concentrations of major gas species (H_2, CH_4, N_2, CO, H_2O, and H_2S). Figure 7 shows equilibrium concentrations of major contaminants in the product gas (H_2S, CS_2, S_2, HCN, and NH_3). These results appear to suggest that product gas major species concentrations do not change significantly above approximately 1000°C, but those for the minor species (contaminants) do. These observations are generally in agreement with TGA test results, as explained earlier.

The recommended Calciner Furnace off-gas composition is as follows:

H_2, vol%	72.99	CS_2, vol%	0.91	C_2H_2, ppmv	111
N_2, vol%	18.29	S_2, vol%	0.20	COS, ppmv	61.7
H_2S, vol%	4.04	HCN, vol%	0.20	NH_3, ppmv	8.7
CO, vol%	3.31	CH_4, vol%	0.05		

Fixed-Bed Testing

A "bulk" sample devolatilization test using 50 grams of the "Shot Coke" material was conducted in GTI's Catalyst Test Facility (CTF, Figure **7**). The CTF is a fixed-bed quartz reactor that enables analysis of the product gas major species using a combination of micro-gas chromatograph and a Fourier Transform Infrared (FTIR) instrument. Unlike the TGA it is not possible to directly measure the change in sample weight over time. The test was conducted during approximately 5 hours in nitrogen, flowing at about 0.6 slpm, at different temperatures ranging from 200 to 1050°C. As shown in Table **2**, the overall weight loss measured during this test was about 10.66%, in general agreement with the TGA test results.

Table 6 compares predicted gas compositions from the HSC Thermodynamic Simulation with the composition of the aggregate gas measured in the fixed-bed test. Because the nitrogen in the CTF includes the N_2 sweep gas, both the CTF and the HSC data are presented N_2-free and normalized.

Table 4. Specifications of Fuel and Nitrogen Input Used for Thermodynamic Simulation

	Hi-S Petcoke							
Ultimate Analysis (As Received)	MW	Ult. Analysis	kg/tonne	kg/tonne (adj1)	kmole/tonne	kmole/tonne (adj1)	**Assumptions**	**Implications to HSC Simulation**
Moisture (107^{o}C), %	18.015	0.66	6.6	6.6	0.366	0.366		
Ash (950^{o}C), %		0.70	7.0	7.0				
Carbon, %	12.011	88.41	884.1	795.7	73.608	66.247	Assume 10% of feed C converted	Input converted C as 50/50 CO/CO2
Hydrogen, %	1.008	3.41	34.1	34.1	33.829	33.829	Assume 100% converted	Input all H as H2(g)
Nitrogen, %	14.007	1.52	15.2	15.2	1.085	1.085	Assume 100% converted	Input all N as N2(g)
Sulfur, %	32.064	5.30	53.0	45.0	1.653	1.403	Assume product contains 1% S and weighs 80% of the feed	Input converted sulfur as S2(g) and ignore residual sulfur in final product
		100.00	1000.00		110.541	102.931		
N2 (100 kg/tonne)	28.014		100		3.570	3.570		

	Lo-S Petcoke							
Ultimate Analysis (As Received)	MW	Ult. Analysis	kg/tonne	kg/tonne (adj1)	kmole/tonne	kmole/tonne (adj1)	Assumptions	
Moisture (107^{o}C), %	18.015	0.66	6.6	6.6	0.366	0.366		
Ash (950^{o}C), %		1.05	10.5	10.5				
Carbon, %	12.011	89.77	897.7	807.9	74.740	67.266	Assume 10% of feed C converted	
Hydrogen, %	1.008	3.71	37.1	37.1	36.806	36.806	Assume 100% converted	Input all H as H2(g)
Nitrogen, %	14.007	1.63	16.3	16.3	1.164	1.164	Assume 100% converted	Input all N as N2(g)
Sulfur, %	32.064	3.18	31.8	27.0	0.992	0.842	Assume product contains 1% S and weighs 80% of the feed	Input converted sulfur as S2(g) and ignore residual sulfur in final product
		100.00	1000.00		114.067	106.443		
N2 (100 kg/tonne)	28.014		100		3.570	3.570		

Table 5. Equilibrium Composition of Calciner Furnace Product Gas at Different Temperature ("Shot Coke")

C:\Documents and Settings\slimane.GTINET\My Documents\2-Rachi						Data:							
	Phase	**Units**	**MW g/mol**	**Min**	**Max**				**Hi-S Petcoke**				
				0.000	100.000	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Temperatu	0	C	0.000	100.000	1500.000	1.00E+02	3.00E+02	5.00E+02	7.00E+02	9.00E+02	1.10E+03	1.30E+03	1.50E+03
C	3	mol-%	12.011	100.000	100.000	1.00E+02	1.00E+02	1.00E+02	1.00E+02	1.00E+02	1.00E+02	1.00E+02	1.00E+02
H2(g)	1	mol-%	2.016	0.060	72.990	5.95E-02	4.95E+00	3.55E+01	6.44E+01	7.04E+01	7.15E+01	7.22E+01	7.30E+01
CH4(g)	1	mol-%	16.043	0.047	54.700	5.47E+01	5.10E+01	2.74E+01	5.52E+00	1.01E+00	2.69E-01	1.00E-01	4.74E-02
N2(g)	1	mol-%	28.013	18.290	29.740	2.97E+01	2.90E+01	2.44E+01	1.98E+01	1.88E+01	1.86E+01	1.85E+01	1.83E+01
H2S(g)	1	mol-%	34.076	4.041	10.150	1.02E+01	9.90E+00	8.33E+00	6.75E+00	6.38E+00	6.16E+00	5.39E+00	4.04E+00
H2O(g)	1	mol-%	18.015	0.001	1.000	5.33E+00	5.11E+00	3.92E+00	9.96E-01	8.38E-02	1.14E-02	2.55E-03	8.07E-04
CO(g)	1	mol-%	28.010	0.000	1.000	3.80E-08	1.42E-03	2.29E-01	2.45E+00	3.29E+00	3.33E+00	3.33E+00	3.31E+00
CS2(g)	1	mol-%	76.131	0.000	1.000	5.04E-12	5.43E-08	5.92E-06	2.53E-04	6.73E-03	7.70E-02	3.84E-01	9.10E-01
S2(g)	1	mol-%	64.120	0.000	1.000	6.66E-14	2.56E-09	5.05E-07	3.04E-05	1.01E-03	1.36E-02	7.67E-02	2.01E-01
HCN(g)	1	mol-%	27.026	0.000	1.000	1.07E-17	3.73E-10	1.35E-06	1.18E-04	1.98E-03	1.44E-02	6.29E-02	1.96E-01
CO2(g)	1	mol-%	44.010	0.000	1.000	1.26E-02	5.93E-02	1.27E-01	5.97E-02	3.00E-03	2.52E-04	3.97E-05	9.62E-06
NH3(g)	1	mol-%	17.030	0.001	1.000	1.35E-02	4.07E-02	4.27E-02	1.67E-02	5.81E-03	2.57E-03	1.39E-03	8.72E-04
C2H2(g)	1	mol-%	26.038	0.000	1.000	1.02E-30	1.05E-17	1.63E-11	3.93E-08	4.82E-06	1.36E-04	1.63E-03	1.11E-02
COS(g)	1	mol-%	60.070	0.000	0.000	5.07E-07	1.15E-04	1.78E-03	8.00E-03	9.28E-03	9.14E-03	8.12E-03	6.17E-03
S(g)	1	mol-%	32.060	0.000	0.000	2.68E-33	1.48E-20	2.41E-14	1.84E-10	1.01E-07	9.43E-06	2.51E-04	2.65E-03
C2H6(g)	1	mol-%	30.069	0.000	0.000	1.08E-05	2.00E-04	3.44E-04	7.48E-05	1.04E-05	2.19E-06	6.76E-07	2.77E-07
C2H4(g)	1	mol-%	28.054	0.000	0.000	2.40E-15	3.59E-09	1.93E-06	2.28E-05	6.02E-05	1.07E-04	1.61E-04	2.24E-04
NO(g)	1	mol-%	30.006	0.000	0.000	1.00E-34	3.42E-26	2.73E-19	1.66E-15	1.56E-13	3.32E-12	3.25E-11	1.92E-10
SO2(g)	1	mol-%	64.059	0.000	0.000	1.30E-18	4.31E-14	7.38E-12	7.63E-11	4.47E-11	2.30E-11	1.27E-11	6.78E-12
O2(g)	1	mol-%	31.999	0.000	0.000	1.00E-34	1.00E-34	2.42E-28	3.39E-23	6.98E-21	2.14E-19	2.76E-18	2.03E-17
S8(g)	1	mol-%	256.480	0.000	0.000	1.21E-26	2.73E-28	1.36E-28	6.00E-27	2.18E-24	2.57E-22	4.32E-21	9.10E-21
NO2(g)	1	mol-%	46.006	0.000	0.000	1.00E-34	1.00E-34	3.97E-34	1.40E-28	5.58E-26	2.79E-24	5.20E-23	5.10E-22
SO3(g)	1	mol-%	80.058	0.000	0.000	1.00E-34	1.29E-29	6.66E-25	1.12E-22	1.22E-22	8.21E-23	5.64E-23	3.62E-23
NO3(g)	1	mol-%	62.005	0.000	0.000	1.00E-34	1.00E-34	1.00E-34	1.00E-34	1.00E-34	1.00E-34	1.00E-34	1.00E-34

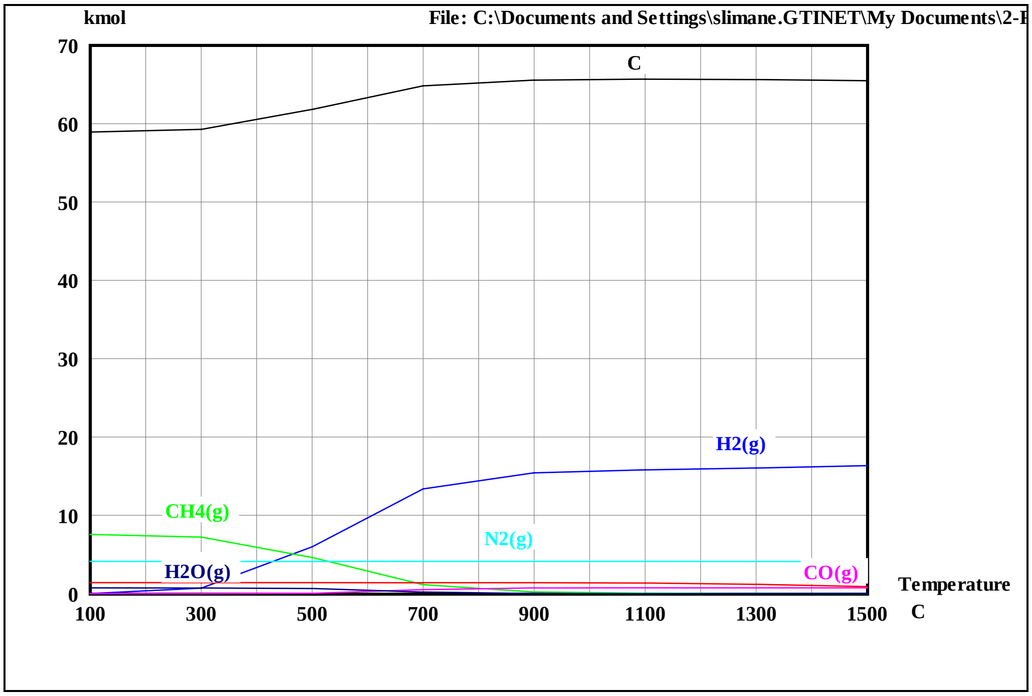

Figure 4. Equilibrium Amounts of Major Calciner Furnace Products at Different Temperatures ("Shot Coke")

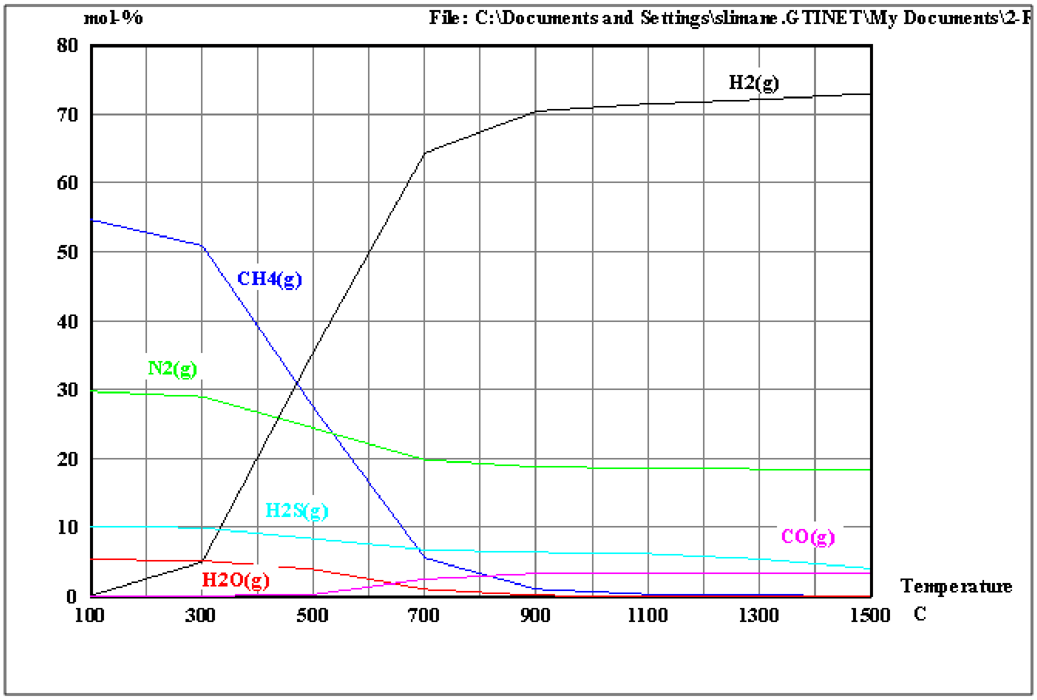

Figure 5. Equilibrium Concentrations of Major Calciner Furnace Product Gas Species at Different Temperatures ("Shot Coke")

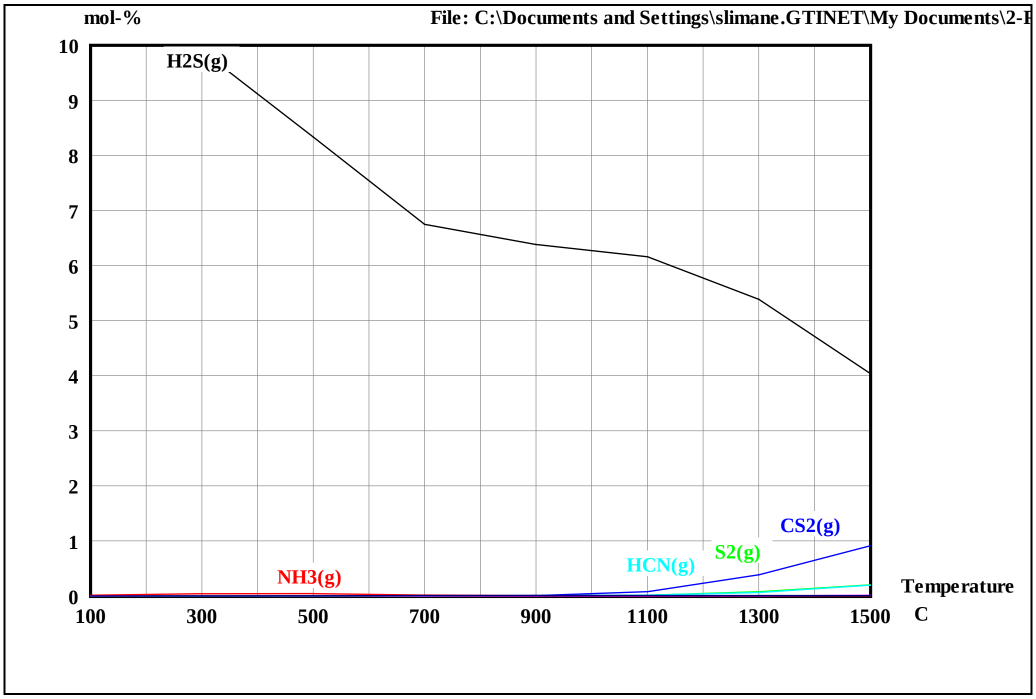

Figure 6. Equilibrium Concentrations of Major Contaminants in Calciner Furnace Product Gas ("Shot Coke")

Table 6. Gas Composition, vol % N_2-Free and Normalized, Measured (CTF) and from Simulation (HSC)

	800-1000°C		1500°C
	CTF	HSC	HSC
H_2	94.22	86.7	89.4
N_2	--	--	--
CH_4	1.14	1.2	0.1
H_2O	3.12	0.1	0.0
CO	0.71	4.1	4.1
H_2S	0.29	7.9	4.9
HHV, BTU/scf	338.9	376.0	353.9

It is possible to estimate[1] the rate of devolatilization of the sample. This estimation is used to make Figure 8, a simulated weight loss profile for the Devolatilization Test. Like in TGA Test No. 2, more material is devolatilized at 600°C than during the other periods. The flat region at the start of 800°C occurred because all flow data were deleted – a large spike of H_2O may have temporarily overwhelmed the GC giving data with nearly no N_2. Variation in the bed temperature at 1050°C is from a poor thermocouple connection; manual readings indicate a constant bed temperature.

Figure 9 confirms the HSC simulations: different compositions are observed at different temperatures, and around 500°C, H_2 surpasses CH_4 as the dominant gas product. Data in the low temperature range may be skewed because of the high moisture content in the sample, which may have been absorbed from the ambient air. One big difference between the two sets of data is the CO concentration. The HSC simulation calculates equilibrium gas composition, but gases created in the CTF are carried away by the N_2 sweep gas and are not given the opportunity to react further. Note that although the moisture concentration is high in the low-temperature setpoints of the CTF test, the total mass lost through about 400°C is only about 1% of the total sample mass.

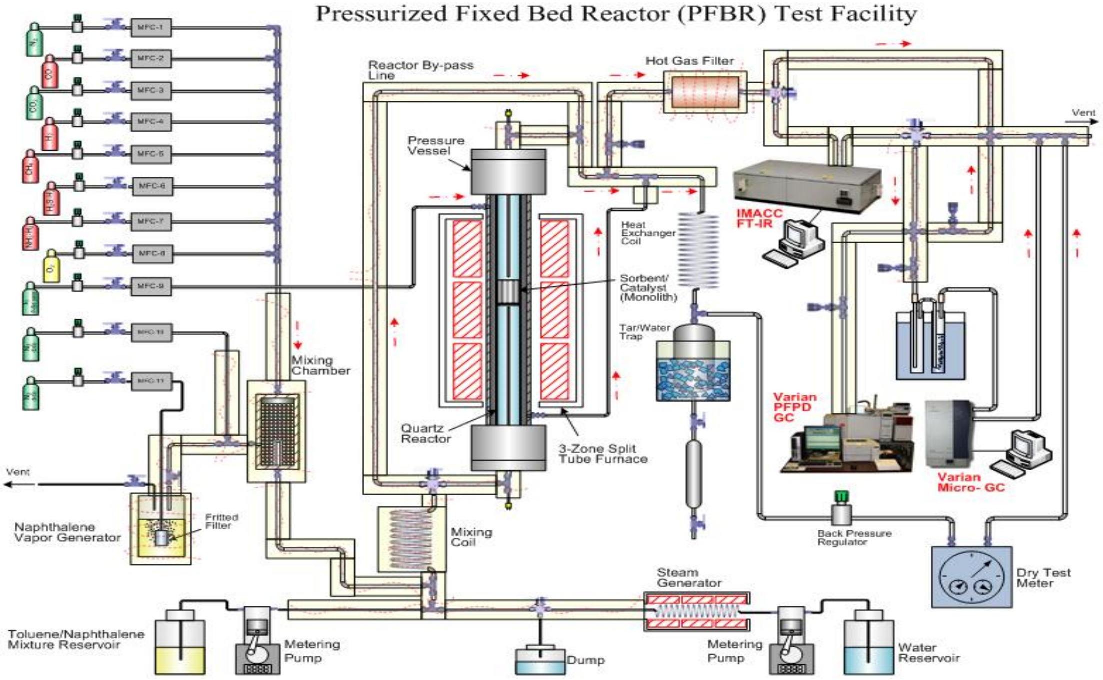

Figure 7. High-Temperature Sorbent/Catalyst Test Facility in GTI's Hot Gas Cleanup Laboratory

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[1] If the N_2 flow is constant at 0.6 slpm and that all N_2 is from the sweep gas, then Total Flow = 0.6 slpm ÷ % N_2 . Then convert volume flow to a mass flow using the gas composition.

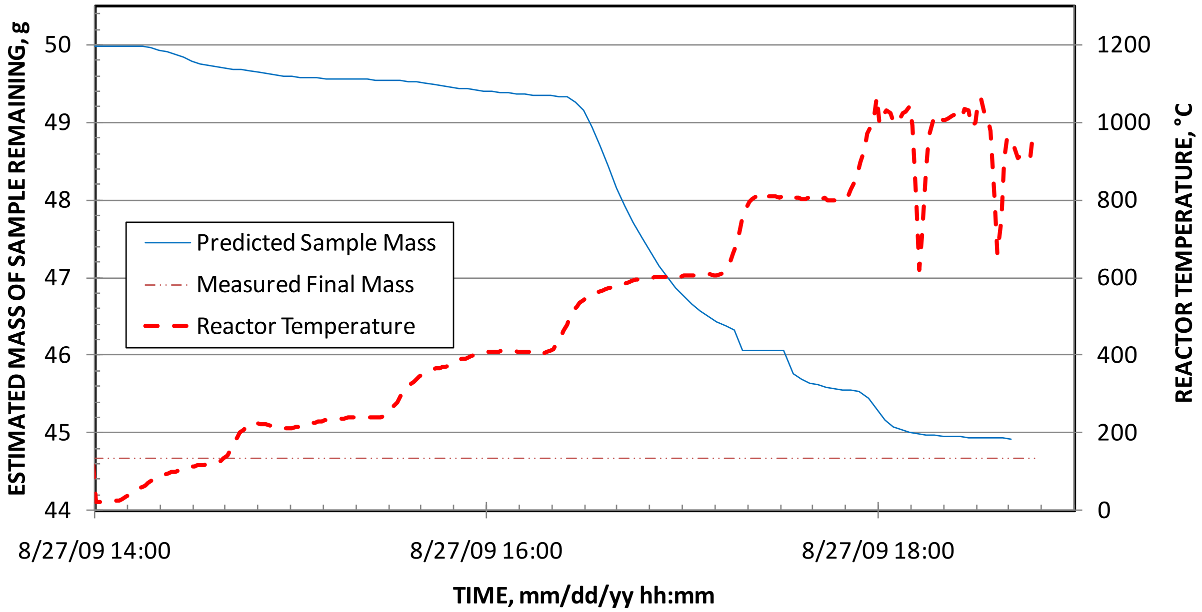

Figure 8. Estimated Mass Loss Curve, Calculated from the Rate of Material Devolatilization[2]

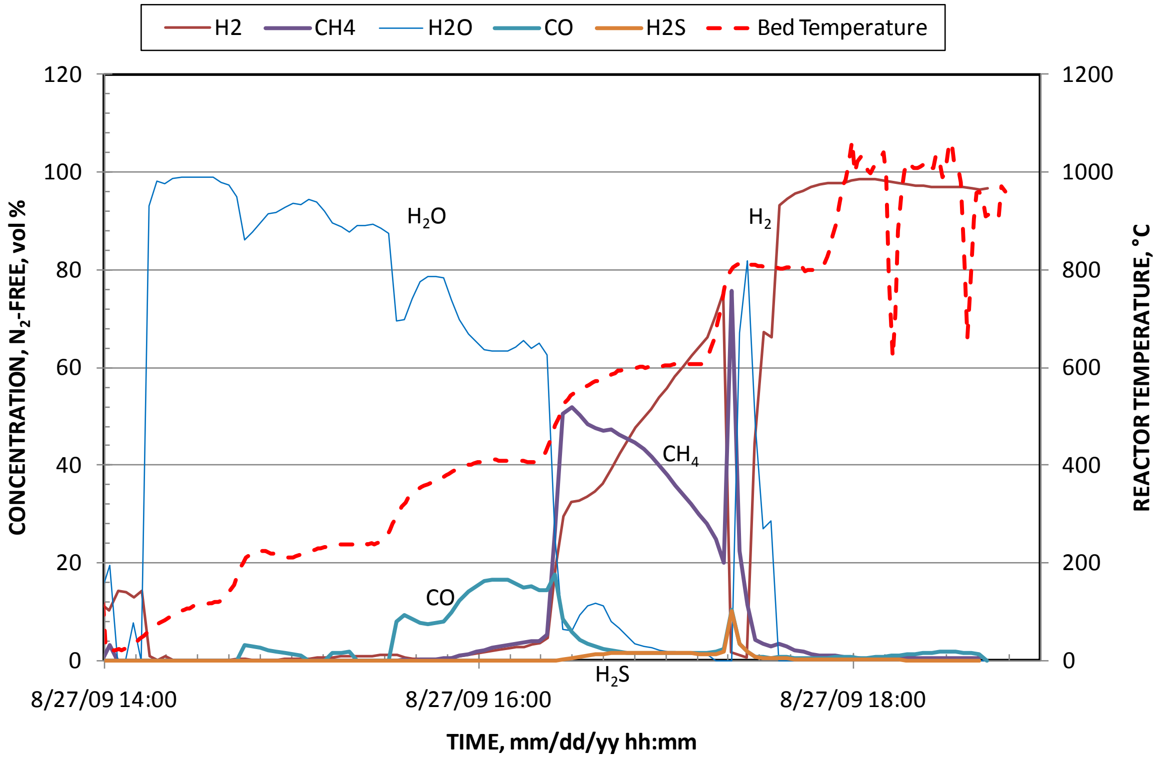

Figure 9. Measured Gas Concentration, N_2-Free and Normalized, in Pyrolysis Test

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[2] Note that variation in the bed temperature at 1050°C is from a poor thermocouple connection; manual readings indicate a constant bed temperature.

NOTE: Results reported by the ECRS Lab on a pyrolysis test conducted at 1100°C using a Green Petcoke sample (type not identified). Since the goal of this work is to determine the composition of gas produced at 1500°C, some virgin material was subjected to a near-instantaneous change directly to 1100°C in the ECRS Lab. The solid devolatilized so quickly that the gas composition was not observed by the Gas Chromatograph, but numerous minor and trace compounds were measured. These are summarized here.

Batch #091238
Sample #091298-001, 'Green' Pet Coke
Sample heated to 1100°C using a pyroprobe and resulting pyrolysate analyzed by GC/MS

	Est Conc. µg/g	Match Quality
1,1'-Biphenyl	3.3	94
1-Methylnaphthalene	4.3	93
2-Ethenylnaphthalene	0.7	91
2-Methylnaphthalene	13.5	91
Acenaphthylene	1.4	87
Anthracene	3.9	93
Benz[a]anthracene	5.2	96
Benzene	89.5	91
Benzo(a)pyrene	1.1	81
Benzo(b)fluoranthene	0.9	93
Benzo(b)naptho[2,3-d]furan	0.7	90
Benzo(e)pyrene	2.0	91
Benzo(k)fluoranthene	0.6	70
Benzonaphthothiophene	16.4	96
Benzonaphthothiophene	4.1	94
Benzonaphthothiophene	5.6	95
Benzonaphthothiophene	0.6	93
Benzothiophene or Benzo(b)thiophene	16.6	91
C19H14, possibly Methylchrysene or Methyltriphenylene	0.9	55
C2-Benzonaphthothiophene	1.8	94
C2-Benzonaphthothiophene	1.1	81
C2-Benzonaphthothiophene	2.1	89
C2-Biphenyl	0.8	87
C2-Biphenyl or methyldiphenylmethane	0.6	86
C3-benzene	2.1	95
C3-benzene	6.4	91
C3-benzene	0.5	91
C3-benzene	3.8	91
Carbazole	4.3	95
Chrysene	10.8	98
coelution of 2 undidentified components, MW 226+, 218+	1.4	--
coelution of 2 unidentified components, MW 242+, 248+	1.4	--
coelution of 3 unidentified components	0.8	--
coelution of Styrene and o-Xylene	6.8	93 / 86
Dibenzofuran	5.0	91
Dibenzothiophene	15.6	97
Dimethylbenzo(b)thiophene	0.6	96
Dimethyldibenzothiophene	2.3	93
Dimethyldibenzothiophene	3.6	80
Dimethyldibenzothiophene	2.5	83
Dimethyldibenzothiophene	1.2	83
Dimethylnaphthalene	3.5	97
Dimethylnaphthalene	3.6	90
Dimethylnaphthalene	0.8	95
Dimethylnaphthothiophene	1.6	90
Dimethylphenanthrene or Dymethylanthracene	1.8	96
Dimethylpyrene or Terphenyl	0.9	93
Ethylbenzo(b)thiophnene	0.8	76
Fluoranthene	3.1	93
Fluorene	4.8	97
Hexamethylcylcotrisiloxane	7.0	90

	Est Conc. µg/g	Match Quality
Indenothienothiophnene or Benzothienobenzothiophene, $C_{14}H_{8}S_{2}$	3.4	94
m,p-Xylene	14.9	97
Methylbenzo(b)thiophene	4.2	90
Methylbenzonaphthothiophene	4.2	93
Methylbenzonaphthothiophene	8.6	95
Methylbenzonaphthothiophene	6.5	83
Methylbenzonaphthothiophene	1.1	80
Methylbiphenyl	1.9	91
Methylbiphenyl	0.4	90
Methylcarbazole	0.8	95
Methylchrysene or Methyltriphenylene	6.4	95
Methylchrysene or Methyltriphenylene	2.5	95
Methyldibenzofuran	3.0	93
Methyldibenzofuran	3.3	93
Methyldibenzofuran	0.5	90
Methyldibenzothiophene	6.6	94
Methyldibenzothiophene	7.9	93
Methylfulorene	1.4	93
Methylfulorene	0.5	97
Methylphenanthrene or Methylanthracene	4.8	93
Methylphenanthrene or Methylanthracene	5.2	83
Methylphenanthrene or Methylanthracene	2.1	90
Methylphenanthrene or Methylanthracene	3.5	92
Methylphenanthrothiophene	2.3	83
Methylphenanthrothiophene	3.9	83
Methylphenanthrothiophene	0.9	78
Methylpyrene or Benzofluorene	1.5	92
Methylpyrene or Benzofluorene	3.3	93
methylpyrene or Benzofluorene	4.9	97
Methylpyrene or Benzofluorene	1.8	93
Methylpyrene or Benzofluorene	0.9	94
MW 262+, possibly a C2-Benzonaphthothiophene	2.5	70
MW 262+, possibly a C2-Benzonaphthothiophene	1.6	60
Naphthalene	29.0	94
Phenanthrene	12.3	95
possibly Phenanthro[4,5-bcd]thiophene	1.2	64
possibly Phenylanthracene	1.2	74
Pyrene	8.5	95
Quinoline or Isoquinoline	1.0	94
Toluene	76.8	91
Unidentified, MW 184+, possibly $C_{12}H_{8}S$ (Naphthothiophene)	0.9	--
Unidentified, MW 196+, possibly $C_{13}H_{12}N_{2}$ or $C_{13}H_{12}Si$	0.8	--
Unidentified, MW 196+, possibly $C_{13}H_{12}N_{2}$ or $C_{13}H_{12}Si$	1.0	--
Unidentified, MW 196+, possibly $C_{13}H_{12}N_{2}$ or $C_{13}H_{12}Si$	1.4	--
Unidentified, MW 202+, most likely $C_{16}H_{10}$	0.7	--
Unidentified, MW 206+	1.1	--
Unidentified, MW 210+	2.8	--
Unidentified, MW 256+	0.8	--
Unidentified, MW 258+	2.0	--
Unidentified, MW 258+	0.6	--
Unidentified, MW 258+	0.7	--
Unidentified, MW 260+	0.5	--

TOTAL	535.3	µg/g fuel
	0.0005353	g/g fuel
	99.95%	fuel not accounted for

APPENDIX B

Aspen HYSYS simulation of ACHP concepts

HYSYS Simulation Models

Models of the steps used to recover heat and power from coke calcination were built using AspenTech's HYSYS program. The latest edition (Version 7.0 and 7.1) of the HYSYS program was used for these studies. The HYSYS program is recognized as an industry standard for doing heat and material balances and describing chemical reactions, and has had thousands of industrial users for over 20 years. Aspen HYSYS is a market-leading process modeling tool for conceptual design, optimization, business planning, asset management, and performance monitoring for oil & gas production, gas processing, petroleum refining, and air separation industries.

Features

Best-in-class physical properties methods and data: Aspen HYSYS offers a comprehensive thermodynamics foundation for accurate calculation of physical properties, transport properties, and phase behavior for the oil & gas and refining industries. The model contains correlations for the enthalpy and entropy of chemical compounds as a function of temperature and pressure. It also adjusts these parameters for non-idealities during the mixing of compounds in actual process streams.
Comprehensive library of unit operation models including distillation, reactions, heat transfer operations, rotating equipment, controller, and logical operations in both the steady state and dynamics environments.

Limitations

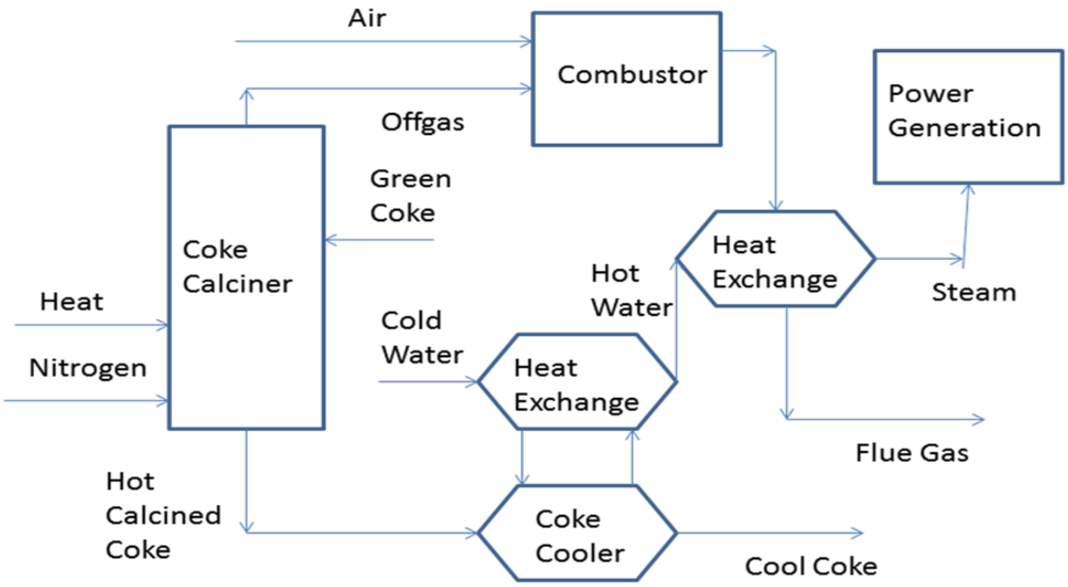
AspenTech's HYSYS product does not have correlations that describe the physical properties of solids, like coke, that contain multiple chemical elements, like carbon, sulfur, hydrogen, etc. Their AspenPlus product does have this capability. In this study, coke was treated in the HYSYS models as pure carbon. If the coke had elemental sulfur content, the sulfur would melt at high temperatures and cause an enthalpy error. When green coke was devolatilized, some of the carbon was converted by a conversion reactor block into methane (volatiles) and hydrogen sulfide (acid gas) for feed in the offgas to the combustion reactions. This had less enthalpy error than other options in the HYSYS model.

The reciprocating internal combustion engine is not one of the process models in the HYSYS system. It was simulated in this model as a combustor (equilibrium reactor) followed by a power generator (reciprocating expander). As explained below, several other process steps were modeled by combinations of several blocks in the HYSYS models. The steady state version of the HYSYS model was used. It is possible to convert this to the dynamic version so that changes that occur with time, like a step change in coke flow rate, can be simulated.

Simple Description of Models

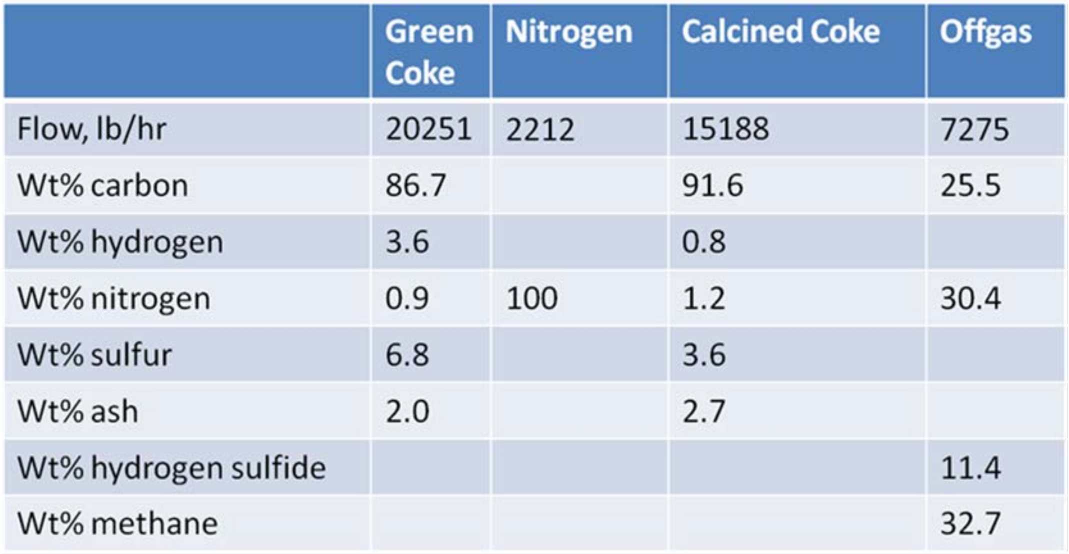
Two major versions of the model were built for this project. Figure 1 depicts the steps in the first model for the Base Case and combined heat and power (CHP) generation. In the Base Case representing normal coke industry operations, no heat or energy recovery steps are included downstream of the
calciner.

Figure 1 CHP Model



In the Base Case (and the CHP variations of this process), green coke, nitrogen gas and heat are added to the coke calciner. The calciner is typically operated at 1500 °C (or 2732 °F) and a typical green coke feed rate of 9,186 kg/hr or 20,251 lb/hr was used in the model. Typical nitrogen purge is 1,004 kg/hr or 2,214 lb/hr. After sufficient time, calcined coke is removed from the bottom of the calciner, while offgas formed from the removal of volatiles from the coke is removed from the top of the calciner. Volatiles removed was 25 wt% of the green coke so the offgas stream is 3300 kg/hr or 7575 lb/hr, including the purge nitrogen. The other components in the calciner mass balance are shown in Table 1. In order to model the physical properties of the calciner streams in HYSYS, the green and calcined cokes were considered to be pure carbon. The offgas was considered to consist of carbon fines, nitrogen gas, and the volatile hydrocarbons were considered to be hydrogen sulfide and methane.

Table 1 - Calciner Mass Balance

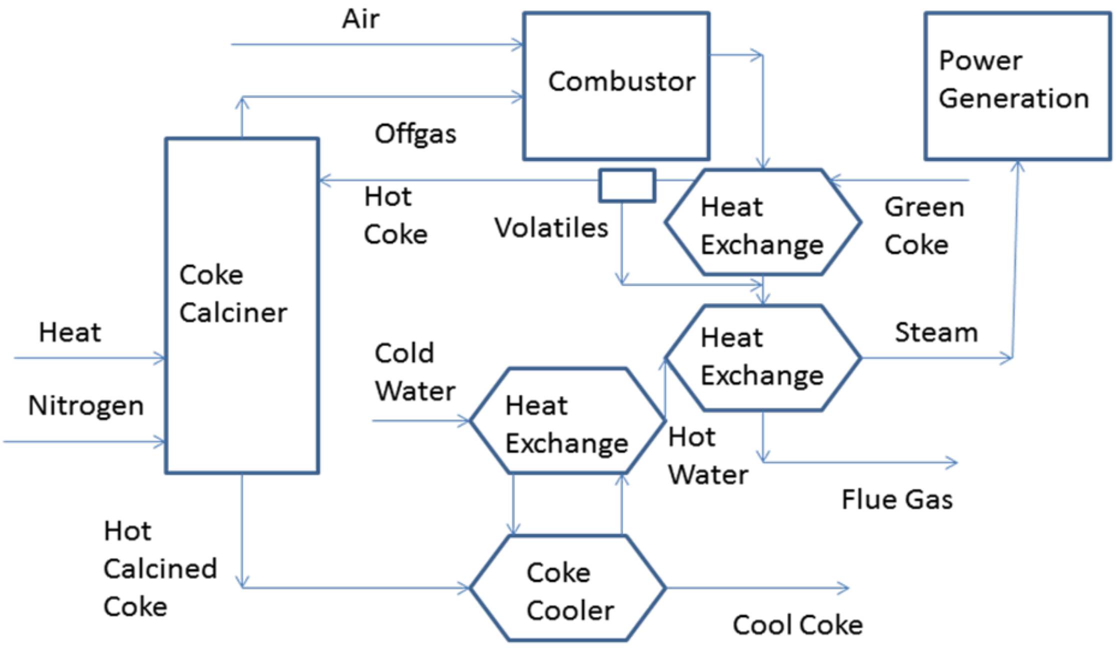


	Green Coke	Nitrogen	Calcined Coke	Offgas
Flow, lb/hr	20251	2212	15188	7275
Wt% carbon	86.7		91.6	25.5
Wt% hydrogen	3.6		0.8	
Wt% nitrogen	0.9	100	1.2	30.4
Wt% sulfur	6.8		3.6	
Wt% ash	2.0		2.7	
Wt% hydrogen sulfide				11.4
Wt% methane				32.7

In order to improve the thermal efficiency of the process with CHP, other steps are then added after the calciner in order to recover the thermal and chemical energy from the calciner product streams. The HYSYS model of the CHP process only includes these downstream process steps and does not include the calciner. The calcined coke and offgas properties were taken from Table 1. As shown in Figure 1, the hot coke is cooled by heat exchange with a circulating water stream, and that water stream is, in turn, used to preheat cold water. Indirect heat exchange is used in order to avoid contamination of the water with chemicals, like benzene, that might be extracted from the hot coke if direct contact were used. The off gas from the calciner is fed to a combustor where it is burned with air. The flue gas composition and extent of reaction is predicted using a HYSYS equilibrium reaction block model. The hot flue gas that results is fed to a heat exchanger where the heat removed is used to convert the hot water from the coke cooling loop into steam. The cooled flue gas is then routed for disposal to a stack at 392 °F. In order to predict the composition of the flue gas to the stack, a HYSYS equilibrium reaction block (not shown in Figure 1) was added and the heat recovery for steam formation was adjusted to cover the heat of reaction for this re-equlibration. The steam formed by these heat exchangers is used to generate power. The major variable studied in this model was the amount of air supplied to the offgas combustor. The quality of the steam to power generation was fixed at 50 atm pressure and 400 °C. The air was modeled as 78.09 mol % nitrogen, 20.95 mol% oxygen, 0.93 mol% argon, and 0.03 mol% carbon dioxide.

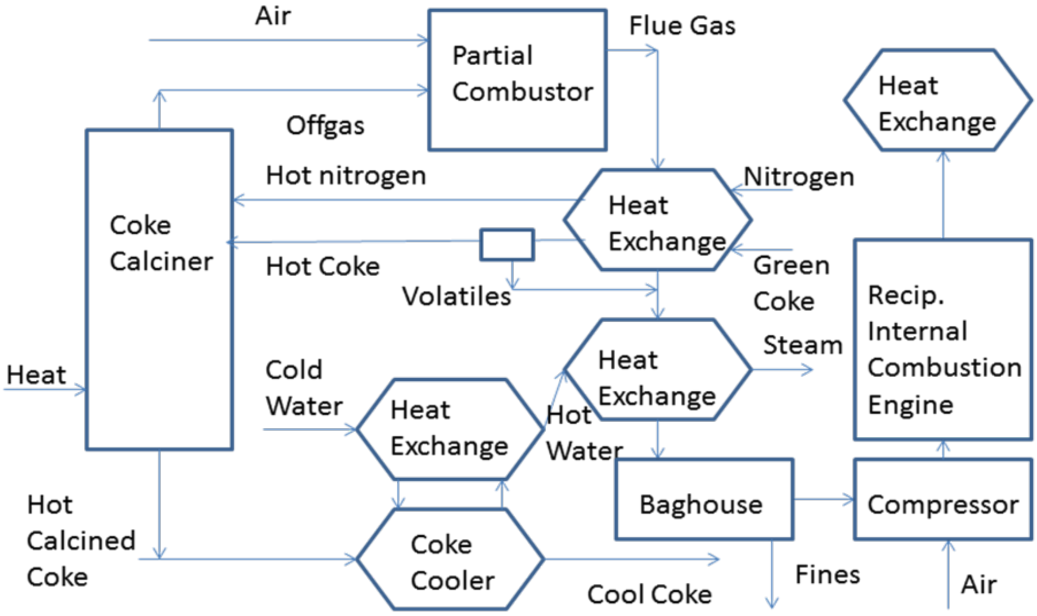
Another variant of this CHP model included adding a direct contact heat exchanger to the flue gas stream in order to preheat and devolatilize the green coke as shown in Figure 2. Any volatiles removed by heating the green coke are separated and added to the flue gas passing through the coke heat exchanger. The major variables in this study included the coke preheat temperature and the amount of air to the offgas combustor. As energy is recovered from the process to preheat and devolatilize the green coke, less external energy needs to be added to the calciner. Coke devolatilization was reported to be 10 wt% from 250-450 °C, 47 wt% from 450-650 °C, and the remaining 43 wt% from 650-1000 °C. The HYSYS model of the hot coke CHP process includes the gasifier and all the downstream steps shown in Figure 2.

Figure 2 Hot Coke CHP Model



The second major HYSYS model built was for steam generation with heat exchange followed by power generation with a reciprocating internal combustion engine (RICE), as shown in Figure 3. It is similar to the CHP case in that the coke is calcined with heat and nitrogen; however all the RICE cases assume preheat of both the green coke and the calincer nitrogen by heat exchange with the hot flue gas. Again, any volatiles removed by preheating the green coke are added to the flue gas that preheats the coke. A major difference from the CHP case is that the air supplied to the offgas combustor is not enough to fully combust the volatiles in the offgas. This is why it is labeled a partial combustor in Figure 3. There is a tradeoff from where the combustion air is added – if at the partial combustor, more steam is produced and if at the RICE, more power is produced. Because not all the carbon particles in the offgas may be gasified in the partial combustor, a baghouse is needed after heat exchange in order to protect the RICE. To use all the fuel energy in the offgas, the cool offgas after heat exchange and the baghouse is combined with more air and compressed for feed to a reciprocating internal combustion engine (RICE). This RICE is used to generate power. The exhaust gas after RICE is fed to a heat exchanger where additional steam can be generated. Major variables in the RICE study include the coke preheat temperature, the order of flue gas exchangers, and the amount of air supplied to the partial combustor and to the RICE.

Figure 3 RICE Model



In each of these studies, adding more heat exchange generally improves the recovery of energy from the process, but increased heat exchanger area adds capital cost.

Results from HYSYS Models

CHP CASES

All the heat from cooling the calcined coke (14.8 MMBtu/hr) was added to preheat the cold water feed from 68 °F to a temperature where the warm water would still be in the liquid phase at 730 psia. Assuming a 5 psi pressure drop in the flue gas exchanger allows the feed to the power recovery turbine to be at 725 psia. The cold water flow rate was adjusted to give a 752 °F steam temperature to the power recovery block. The feed water pump requires 83-85 hp for the cases in Table 2. The power needed to recirculate the water in the coke heat recovery section was less than 1 hp.

Table 2 shows the effect of adding varying amounts of air to the offgas combustor. The maximum power is produced around 10% excess air addition. Also, for each case, about 76 thousand pounds of saturated 50 psia steam is produced at the exhaust of the power generation turbine. The net power production is 84-86 hp less than shown in the table because of pump power requirements.

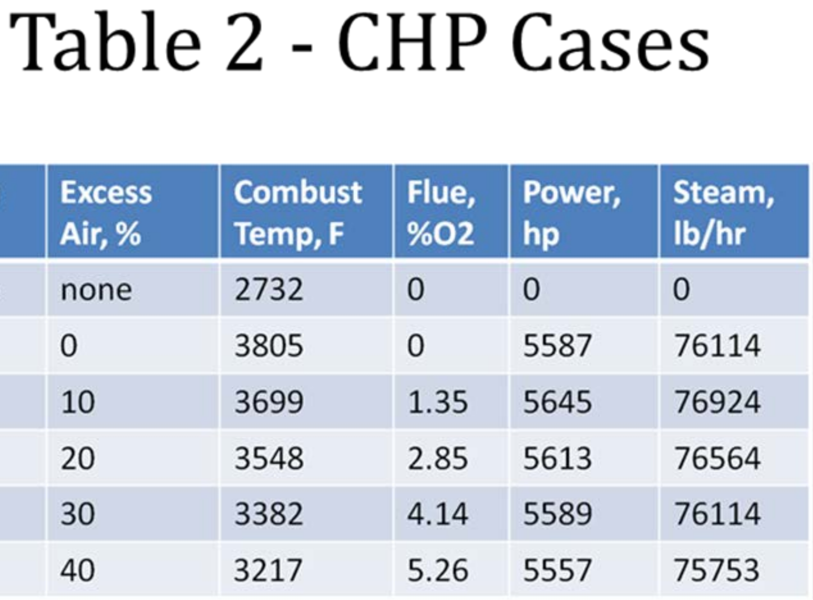


Table 2 - CHP Cases

Case	Excess Air, %	Combust Temp, F	Flue, %O2	Power, hp	Steam, lb/hr
Base	none	2732	0	0	0
1	0	3805	0	5587	76114
2	10	3699	1.35	5645	76924
3	20	3548	2.85	5613	76564
4	30	3382	4.14	5589	76114
5	40	3217	5.26	5557	75753

HOT COKE CHP CASES

The results from the hot coke CHP cases are shown in Table 3. The first hot coke CHP case does not actually heat the coke, but is included for comparison with the corresponding CHP case #2. Because of the energy (enthalpy and entropy) differences as the coke (presumed as pure carbon in the model) is decomposed into the volatiles and calcined coke streams, and as carbon is converted into methane and hydrogen sulfide in the volatiles, there is a slight difference between CHP case #2 (where the coke decomposition is not modeled in HYSYS) and hot coke CHP case #1 (where the decomposition is modeled). The feed water pump requires 80 hp for this case and the circulating water pump requires less than 1 hp.

Table 3 – Hot Coke CHP Cases

Case	Hot coke Temp, F	Excess Air, %	Combust Temp, F	Flue, %O2	Power, hp	Steam, lb/hr	Calciner Q, MMBtu/hr
Base	100	none	2732	0	0	0	21.1
1	100	10	3665	1.74	5639	76800	21.1
2	752	0	3787	0	5317	72400	17.1
3	752	10	3665	1.12	5425	73900	17.1
4	752	20	3504	2.64	5397	73500	17.1
5	1112	40	3164	0	4886	66600	12.4
6	1112	60	2866	0.97	5166	70400	12.4
7	1112	80	2617	3.01	5125	69900	12.4
8	1472	100	2404	0	4802	65400	9.0
9	1472	120	2227	0.78	4982	67900	9.0
10	1472	140	2076	2.32	4955	67500	9.0

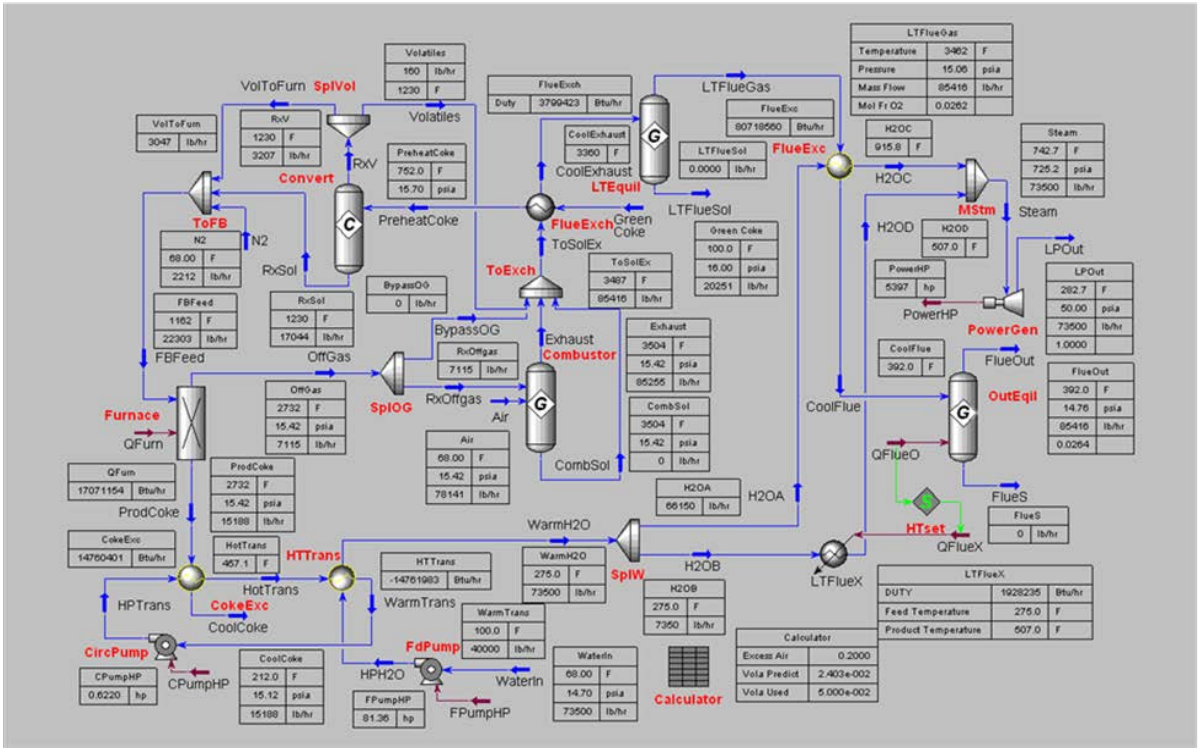
When the coke is preheated to 400 °C (752 °F), 5 wt % of the volatiles are removed in the heating process and transfer to the flue gas stream. The maximum power generation and by-product steam production occur when about 10% excess air is used, based on the remaining offgas fed to the combustor. Although these maximum values are less than when the coke was not preheated, the coke preheat lowers the heat demand on the calciner by 4 MMBtu/hr.

When the coke is preheated to 600 °C (1112 °F), 50 wt % of the volatiles are removed in the heating process and transfer to the flue gas stream. The maximum power generation and by-product steam production occur when about 60% excess air is used, based on the remaining offgas fed to the combustor. Because the excess air calculation is based on the fuel gas going to the combustor, the excess air level is higher in cases #5-7 than in cases #1-4 since a half of the volatiles are added to the flue gas stream downstream of the combustor. These volatiles are combusted in the downstream equilibration steps so that all of the off gas fuel value possible is recovered. Although these maximum power and steam production values continue to decrease with the amount of coke preheat, the extra coke preheat lowers the heat demand on the calciner by 8.7 MMBtu/hr from that required with no coke preheat.

When the coke is preheated to 800 °C (1472 °F), 77 wt % of the volatiles are removed in the heating process and transfer to the flue gas stream. The maximum power generation and by-product steam production occur when about 120% excess air is used, based on the remaining offgas fed to the combustor. Although these maximum power and steam production values continue to decrease with the amount of coke preheat, the extra coke preheat lowers the heat demand on the calciner by 12.1 MMBtu/hr from that required with no coke preheat.

The best process conditions for these cases seemed to be at about 1% oxygen in the flue gas, regardless of the coke preheat temperature. The actual HYSYS hot coke CHP model process flow diagram (PFD) is shown in Figure 4 for case #4 and is much more complex than the block diagram shown in Figure 2. The PFD shows the temperatures, pressures, and flow rates for the streams in the model and the heat transfer duties for the heat exchangers.

Figure 4 – HYSYS Hot Coke Model PFD



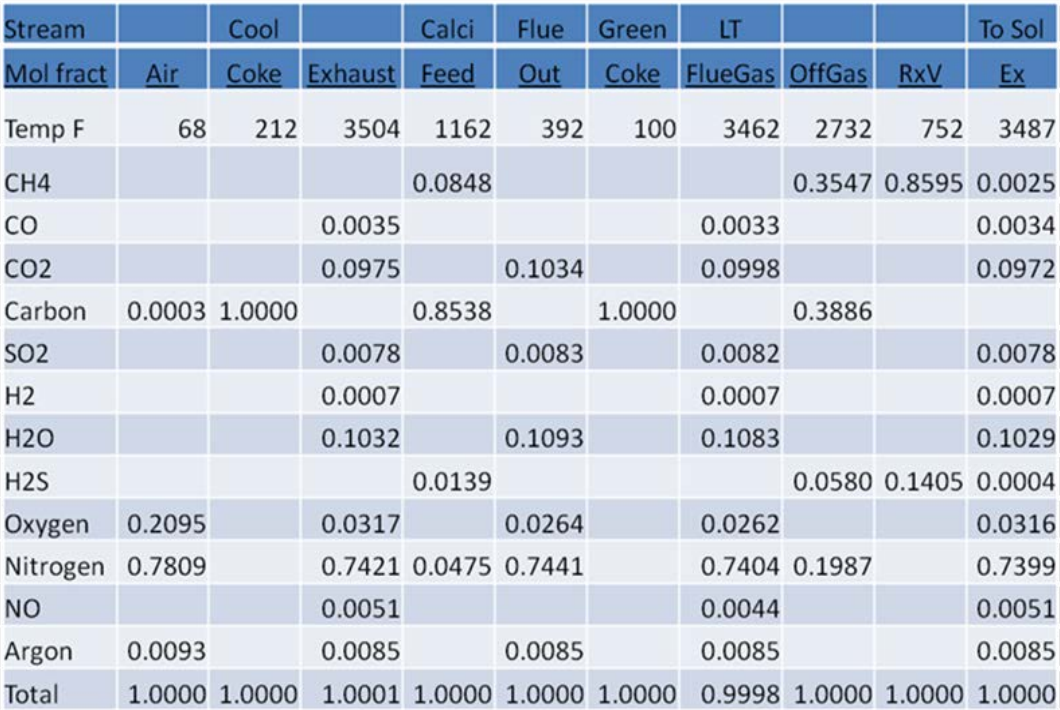
Green coke enters just above the center of the PFD at 100 F and is heated in block FlueExch to 752 F. The Convert block turns the green coke into calcined coke by turning 25 wt% into volatiles (methane and hydrogen sulfide) for routing into the RxV stream and the unconverted carbon is routed to the RxSol stream. Because of the enthalpy change when carbon in the green coke is converted into other compounds, the HYSYS model assumes that the temperature of the product streams are changed. This imputed temperature change is kept in the model in order to keep the model in enthalpy balance. The SplVol block then routes only some of the volatiles to the flue gas in the Volatiles stream since the coke preheat temperature is less than the 2732 F at while full volatiles removal is achieved. The volatiles are transferred to the flue gas stream using the ToExch mixing block. In this case, 5% of the volatiles are released at 752 F and is entered into the HYSYS model via the Calculator block shown at the bottom of Figure 4. The remaining volatiles that would be released at higher temperatures are sent to the VolToFurn stream. The combination of the FlueExch, Convert, SplVol and ToExch blocks in the HYSYS model are equivalent to the green coke preheat exchanger in Figure 2. Block ToFB combines the VolToFurn volatiles with the unconverted coke and the purge nitrogen streams to form the FBFeed stream. Block Furnace then heats this stream to 2732 F and determines that 17.1 MMBtu/hr are required. The Furnace block also separates the feed into the Offgas gas stream and the ProdCoke solid stream. The combination of the ToFB and Furncae blocks in the HYSYS model is equivalent to the Coke Calciner in Figure 2.

Heat exchanger CokeExc cools the calcined coke to 212 F and is equivalent to the Coke Cooler block in Figure 2. The SPlOG allows some Offgas to bypass the combustor, but this capability was not used in any of the cases reported here. The Combustor block thakes the fuel feed and a feed air stream and reacts them to equilibrium. The Calculator block determines the air flow rate to the Combustor as a function of excess air to the fuel stream (RXOffgas) to the combustor. The products from the Combustor are the gaseous Exhaust stream and an unconverted CombSol solids stream, although this solids stream usually has zero flow rate. The ToExch block combines the Combustor product stream with any volatiles from the coke preheat for routing to the FlueExch heat exchanger. The LTEquil block determines the equilibrium concentrations in the flue gas after the temperature has been reduced by green coke preheat and allows any unconverted solids (usually zero) to be removed via the LTFlueSol stream. The LTFlueGas stream is routed to the FlueExc block where stream is generated by reducing the flue gas temperature to 392 F in the CoolFlue stream. The OutEqil block determines the equilibrium concentrations in the flue gas at this temperature and determines the enthalpy change needed for this in theQFlueO heat stream. Stream FlueOut is the final flue gas product to the stack at 392 F and its equilibrium concentrations.

The water for steam production enters the PFD as the WaterIn stream at the center of the bottom of Figure 4. FdPump block raises the pressure to 735 psia and determines that 81 hp are needed. The CIrcPump pump block with heat exchangers CokeExc and HTTrans are equivalent to the cold water heat exchange block in Figure 2 and transfers the heat from cooling the calcined coke into the water feed stream, stream WarmH2O. The SplW blocks routes some of the warm water to the FlueExc heat exchanger and some to the LTFlueX heat exchanger when the heats of forming equilibrium concentrations are collected and the water streams are then combined in block MStm to produce the Steam feed to the power generation step. This steam is at 725 psia and about 750 F in all cases in this study. The PowerGen block then expands this steam to 50 psia and predicts that 5397 hp is generated. The power generation step assumes 75% adiabatic efficiency, or 72.6% polytropic efficiency.

Tabulation of flow rates for all streams in lb/hr, mol/hr and Btu/hr is available from HYSYS but is not presented in this report. Most of these values are presented on the PFD. The compositions of key streams from the HYSYS results are shown in Table 4. Although the NOx content of the Flue Out stream is predicted to be zero at the outlet conditions, the NOx content for the higher temperature streams are 4,000-5,000 ppm and may represent an emissions permitting issue. As coke preheat increases, the peak combustion gas temperature decreases, as shown in Table 3, so the peak NOx levels decrease with the amount of coke preheat.

Table 4 – Hot Coke CHP Case #4



Stream		Cool		Calci	Flue	Green	LT			To Sol
Mol fract	Air	Coke	Exhaust	Feed	Out	Coke	FlueGas	OffGas	RxV	Ex
Temp F	68	212	3504	1162	392	100	3462	2732	752	3487
CH4				0.0848				0.3547	0.8595	0.0025
CO			0.0035				0.0033			0.0034
CO2			0.0975		0.1034		0.0998			0.0972
Carbon	0.0003	1.0000		0.8538		1.0000		0.3886		
SO2			0.0078		0.0083		0.0082			0.0078
H2			0.0007				0.0007			0.0007
H2O			0.1032		0.1093		0.1083			0.1029
H2S				0.0139				0.0580	0.1405	0.0004
Oxygen	0.2095		0.0317		0.0264		0.0262			0.0316
Nitrogen	0.7809		0.7421	0.0475	0.7441		0.7404	0.1987		0.7399
NO			0.0051				0.0044			0.0051
Argon	0.0093		0.0085		0.0085		0.0085			0.0085
Total	1.0000	1.0000	1.0001	1.0000	1.0000	1.0000	0.9998	1.0000	1.0000	1.0000

RICE CASES

No Partial Combustor Air -The HYSYS model for the RICE cases is shown in Figure 5. The first series of cases studied routed all of the air to the RICE unit and none to the partial combustor. The green coke and calciner nitrogen steams were heated to equal temperatures in exchangers Flue1 and N2Ht, and any volatiles produced by the coke heating was separated in block Convert and routed back to the flue gas stream via blocks SplVol and ToExch.

The caliner is represented by the Furnace block where all the feeds are heated to 2732 F. Water1 is used to cook the calcined coke to 300 F and is converted to saturated 150 psi steam. The offgas from the caliner is bypassed around the partial combustor block when no air is fed to it. The baghouse was specified to operate at 300 F, so any heat not used to preheat the calciner feeds was used to heat the Water 3 stream into saturated 150 psi steam in heat exchanger block Flue2. The baghouse removed 1855 lb/hr of coke particles when no air was fed to the partial combustor. The baghouse gas, stream BHGas, was mixed with the air feed to the RICE unit, stream RICEAir, and compressed to 220.4 psia and the block RICE allows the fuel and air to react to equilibrium. The hot gas then flows through an expender, block RICEWk, where power is generated. The Water 2 stream then cools the expander gas to 300 F and generates saturated 150 psi steam. All the 150 psi steam streams are combined into stream Steam. Since the conditions at the baghouse are the same regardless of preheat alternatives, the performance of the upper right part of Figure 5 is independent of amount of preheat used. Table 5 shows the amount of power and steam generated by RICE as a function of the amount of excess air supplied.

The maximum net horsepower produced occurs at about 20% excess air while the maximum water use, corresponding to steam make from the RICE, occurs at about 30% excess air.

Table 6 shows the effect of varying the calciner preheat temperature, with the excess air to the RICE unit fixed at 20%. As the preheat temperature increases, the amount of water fed to the flue gas exchanger, corresponding to steam production, decreases. The final column in Table 6 shows that the total steam production decreases as the preheat is increased. However, the amount of heat needed by the calincer decreases. The optimum case will depend on the value of the byproduct steam versus the cost of fuel gas for the calciner.

Figure 5 – RICE Case

(no partial combustor air)

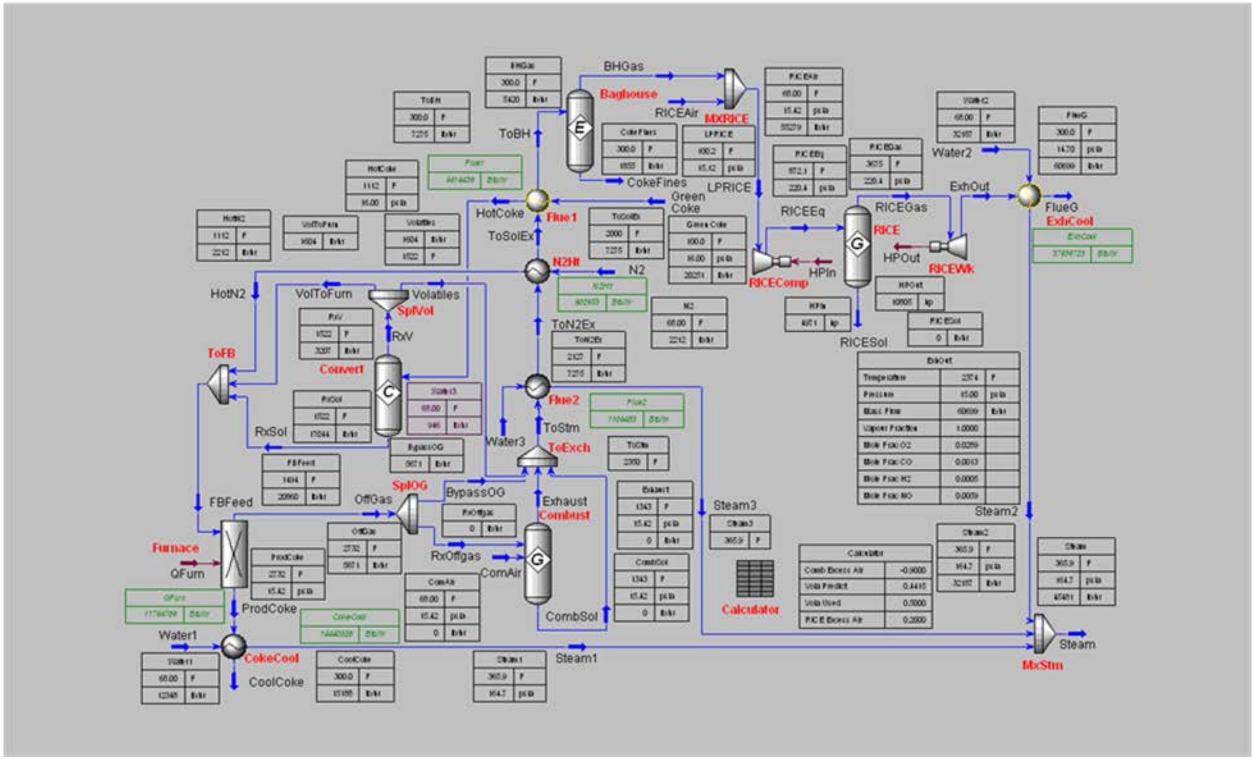


Table 5 – RICE

(no partial combustor air)

Excess Air	Water 2, lb/h	Compress hp	Work hp	Net hp
0%	31292	4270	9984	5714
10%	32038	4260	10450	5830
20%	32187	4971	10805	5834
30%	32199	5321	11122	5801
40%	32174	5671	11422	5751
50%	32141	6021	11711	5690

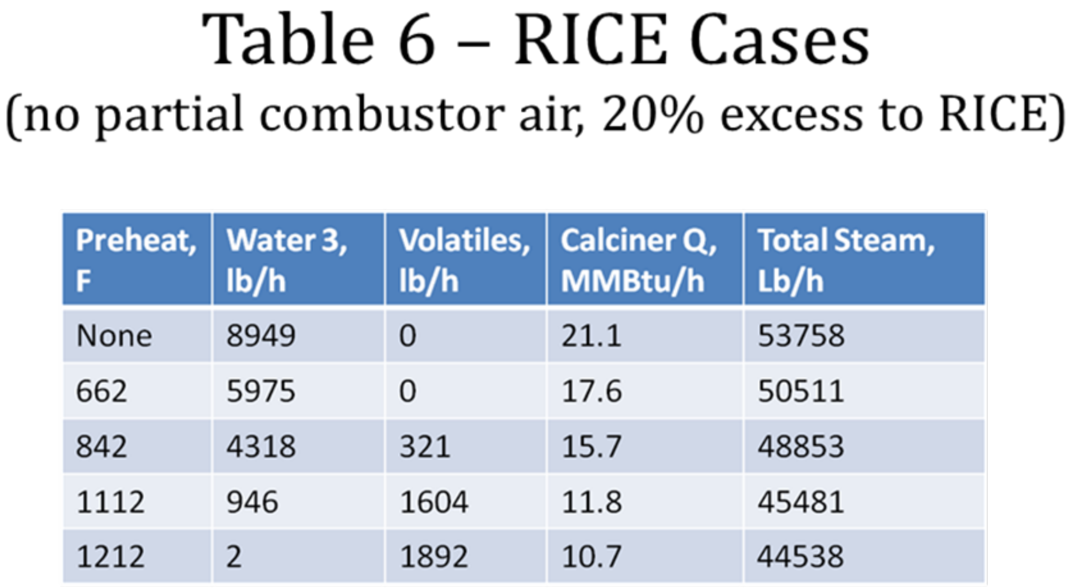


Table 6 – RICE Cases

(no partial combustor air, 20% excess to RICE)

Preheat, F	Water 3, lb/h	Volatiles, lb/h	Calciner Q, MMBtu/h	Total Steam, Lb/h
None	8949	0	21.1	53758
662	5975	0	17.6	50511
842	4318	321	15.7	48853
1112	946	1604	11.8	45481
1212	2	1892	10.7	44538

With Partial Combustor Air – If the stoichiometric amount of air, or more, were supplied to the partial combustor, no fuel value would remain to use in the RICE. Therefore, all cases studied had much less than the stoichiometric amount of air added to the partial combustor. With little air addition, the partial combustor acts like a reformer with substantial production of unsaturated hydrocarbons, if equilibrium occurs in the partial combustor. Figure 6 shows that with 674 lb/hr, or 1% of the stoichiometric amount of combustion air added (or -99% excess air), the adiabatic temperature of the offgas drops from 2732 F to 1171 F, and the product gas has 21% ethylene, 12% propylene, and 7% acetylene. However, all the carbon is converted to gaseous products so no char removal is needed before the RICE. As more air is added, the equilibrium temperature rises, and the amount of acetylene and olefins (ethylene and propylene) decrease. It takes over 20,000 lb/hr of air addition before all unsaturated hydrocarbons are eliminated and the first carbon dioxide formation is seen. Figure 7 shows the overall production of power and steam from the RICE process with partial combustion when neither coke nor nitrogen preheat was done. Because the temperature of the offgas drops with low extent of partial combustion, less heat is available with which to preheat the feeds. Because all of the carbon in the feed is oxidized in either the partial combustor or the RICE combustor, and no coke fines are discarded, more power is produced in the RICE cases with partial combustion (Figure 7) than in the cases without partial combustion (Tables 2, 3, and 5), and similar amounts of stream are produced in all cases. One disadvantage of the partial combustion cases with no preheat is that the full amount of heat (21.1 MMBtu/hr) needs to be supplied to the coke calciner in these cases.

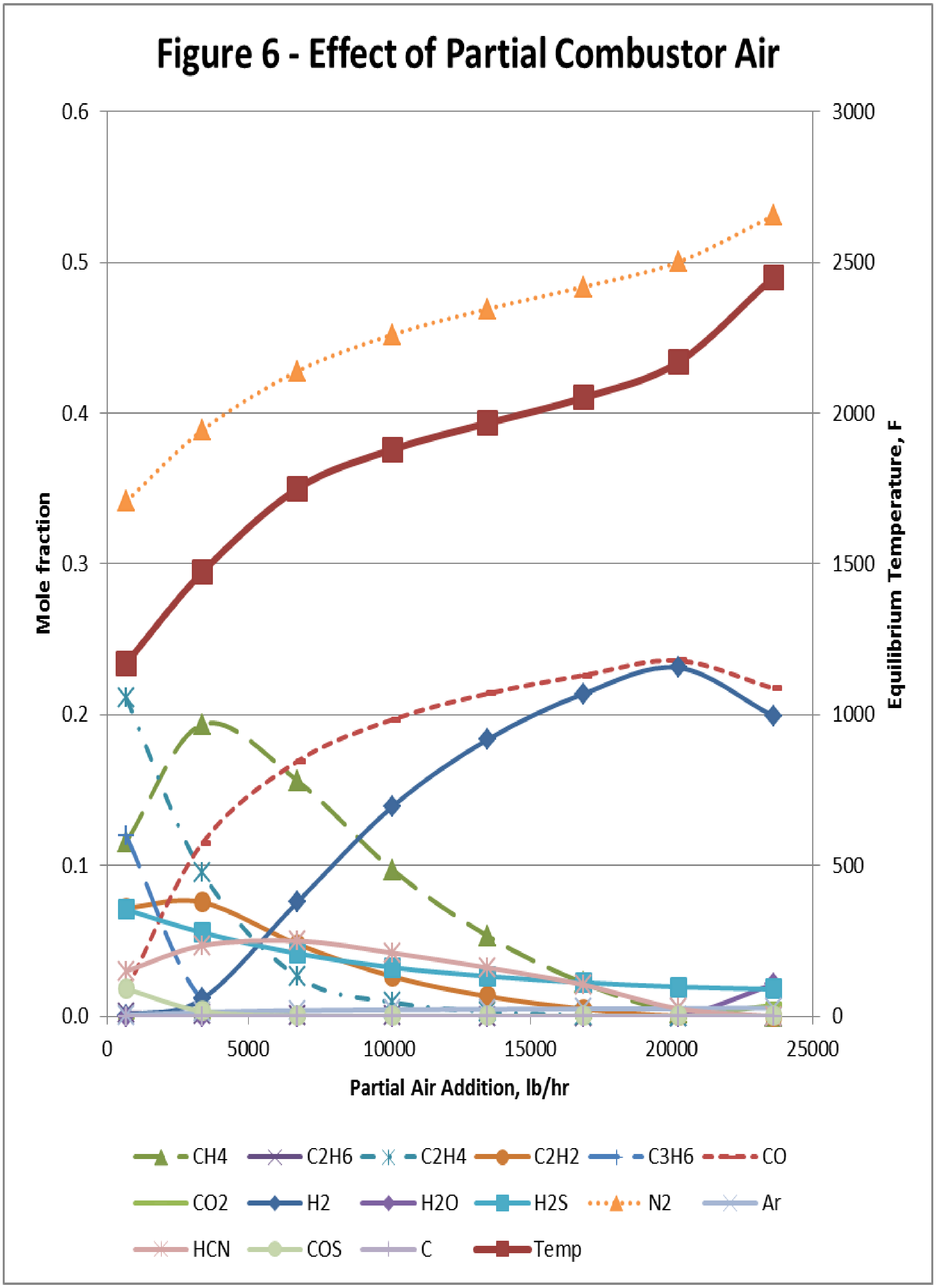


Figure 6 - Effect of Partial Combustor Air

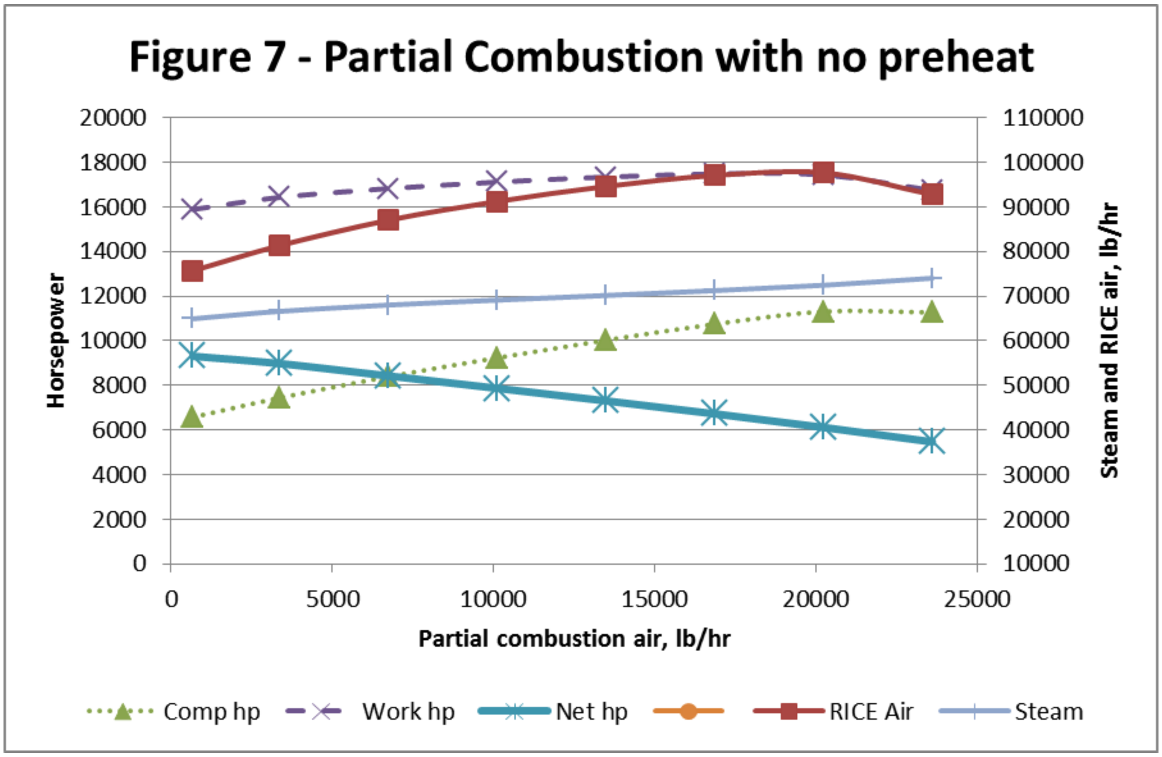


Figure 7 shows that the most power is produced when low amounts of partial combustion air are supplied, but that steam production increases as the amount of partial combustion air is increased. The optimal case will depend on the value for byproduct steam versus byproduct power.

APPENDIX C

Modeling of the Combustion of Off-Gas in a Water-Cooled Cyclone Chamber

[Technical Summary Report by University of Southern California under subcontract S136]

1. Reactor Model:

The water-cooled cyclone chamber is modeled as a reactor cascade consisting of a continuous, stirred-tank reactor (CSTR) followed by two plug-flow reactors (PFR) in series (Fig. 1 shows a schematic of the reactor cascade representing the cyclone chamber). In this model, the off-gas and the primary air are first fed into the CSTR, the exit stream from the CSTR is then fed into the first PFR. Finally, the products from the first PFR are mixed with the secondary air, and then enter the second PFR. Further justification for the formulation of model is provided below:
1: The swirl-vanes in the cyclone chamber are there to introduce turbulence in the flow in order to rapidly mix the off-gas entering the cyclone chamber with the air. Assuming that the swirl-vanes function as intended (i.e., to efficiently and rapidly mix the off-gas with air), it is then reasonable to model that region of the cyclone chamber as a continuous stirred-tank reactor, using the classical equations which predict the time-averaged properties of the CSTR [1].
2: The two-stage cyclone combustion zone can be assumed to function as two plug-flow chemical reactors in series. In our simulations we utilize the classical plug-flow reactor model [1], which assumes a non-dispersive, one-dimensional flow of the chemically reacting gas mixture through the reactor.

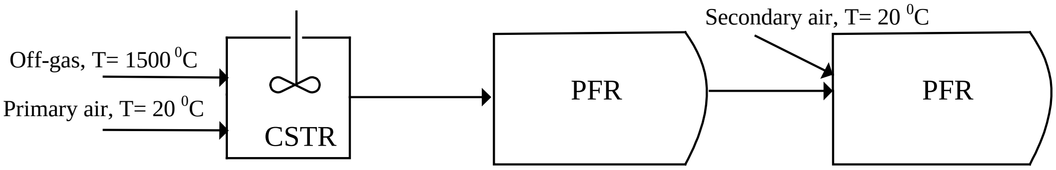


Figure 1. Simplified reactor model of the water-cooled cyclone chamber

Though the reactor cascade model of the cyclone chamber appears reasonable, we recommend that it is verified further experimentally, e.g., through tracer flow experiments.

2. Kinetic Model:

In the simulations we have utilized the following kinetic model in order to describe the combustion of the various components of the off-gas:

(1): For the carbon (C) particle combustion, the following three global gas-solid reactions are used [2-4]. For the frequency factors for all three reactions (and the activation energy for reaction 3) we used values from [2], while for the activation energies for reactions (1) and (2) we used the values reported in [3, 4].

$$C(s) + O_2 \rightarrow CO_2 \qquad (1)$$

$$C(s) + CO_2 \rightarrow 2CO \qquad (2)$$

$$C(s) + 1/2\,O_2 \rightarrow CO \qquad (3)$$

(2): For the combustion of H_2, CO, and CH_4, the GRI 3.0 kinetic model is used [5]. (3): For the combustion of the gas-phase hydrogen sulfide (H_2S), the oxidation mechanism from [6] is used.

The physical size of the reactors, as provided by GTI, and the various reactant flows are shown in Table 1. For these simulations, as recommended by GTI, the heat loss is assumed to be 10% of the total heat generated by the exothermic reactions.

Table 1. Dimensions of the reactors and the size of reactant flows

	Axial length(m)	Diameter(m)	Mass flow rate(g/s)				Inlet particle mass density (kg/m^3)
CSTR	0.276	0.5	Air	O_2 (%)	0.21	Off-gas	0.037198
				N_2 (%)	0.79		
			520.5			67.94	
PFR(1st)	0.924	0.5					
PFR(2nd)	0.56	0.5	Air	O_2 (%)	0.23 (enriched)		
				N_2 (%)	0.77		
			433.75				

<u>Note</u>: Inlet particle mass density = $m_{particle}/V_{off\text{-}gas}$; $V_{off\text{-}gas} = n$ RT / P; $n = m/MW_{ave}$;
$MW_{ave} = MW_{H2} * MF_{H2} + MW_{CH4} * MF_{CH4} + MW_{CO} * MF_{CO} + MW_{H2S} * MF_{H2S} + MW_{N2} * MF_{N2}$
$m_{particle}$: mass of the carbon particles; $V_{off\text{-}gas}$: Volume of the off-gas;
MW_i: molecular weigh of species i; MF_i: molar fraction of species i

Note that, as it became evident during the technical literature search, very little reliable information exists about the kinetics of carbon particle combustion. We strongly recommend, as a result, that a companion investigation is initiated to study the combustion kinetics of carbon particles with the exact physical and chemical characteristics as those encountered with this particular off-gas; it is well-known, that such global kinetic rates are notoriously dependent on the size, shape and surface characteristics of such particles, as well as their chemical composition.

3. Results and Discussion:

CSTR:

Simulations indicate that virtually complete conversion of all the gaseous, off-gas components occurs, as shown in Table 2. The calculated exit temperature of the reactor is 2413 K.

Table 2. Mole fractions of gas-phase components in the inlet and outlet of the CSTR

Mole fraction in the gas phase (%)		H_2	CH_4	CO	H_2S	CO_2	H_2O	N_2	O_2
Inlet	Off-gas	74.61	0.09	1.92	6.32	0	0	17.06	0
	Air							79	21
Outlet		0.4	0	0.6	0	4.8	17.83	70.82	2.4

In the CSTR (mixing) part of the cyclone chamber, the calculated conversion of the carbon particles is 35.2%. Specifically, the inlet C mass flow is 24.2 g/s, while the outlet mass flow is 15.7 g/s.

PFR (1st):

In the first PFR, the calculated additional conversion of the carbon particles is 31.8%. Specifically, the inlet C mass flow is 15.7 g/s (the exit flow from the CSTR), while the outlet mass flow is 10.7 g/s, as Fig. 2 below indicates.

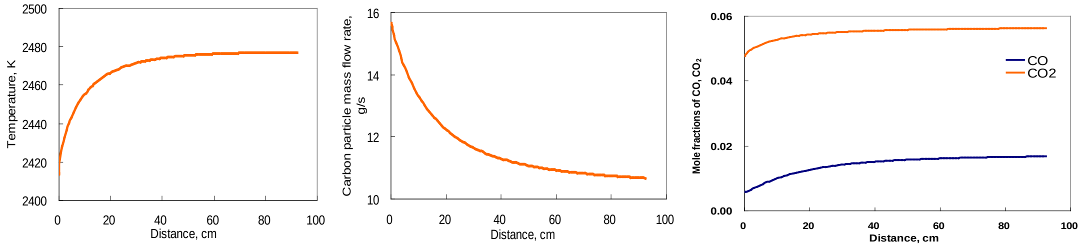


Figure 2. Results from the first PFR

PFR (2nd):

In the second reactor our simulations indicate that virtually all the CO was converted into CO_2 (see Fig. 3). The conversion of carbon particles is 72%. The inlet C mass flow is 10.7 g/s (the exit flow from the 1st PFR), while the outlet mass flow is 3.0 g/s. The total conversion of the carbon particles in the cascade of these three reactors (representing the water-cooled cyclone chamber) is 87.6%. It must reiterated here that these simulations are very much dependent on the carbon particle global kinetic rate parameters (i.e., activation energies and pre-exponential factors). As reported in one of the Quarterly Reports, when using activation energies for the carbon particle combustion rates from the literature derived with larger carbon particles [2], the combustion in the water-cooled cyclone chamber consumed a significantly lower fraction of the carbon particles than it has here.

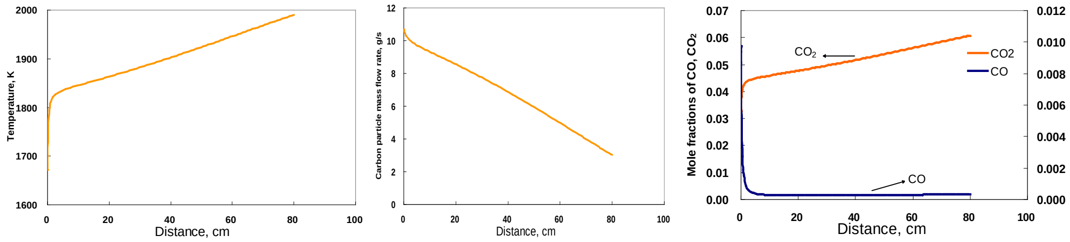
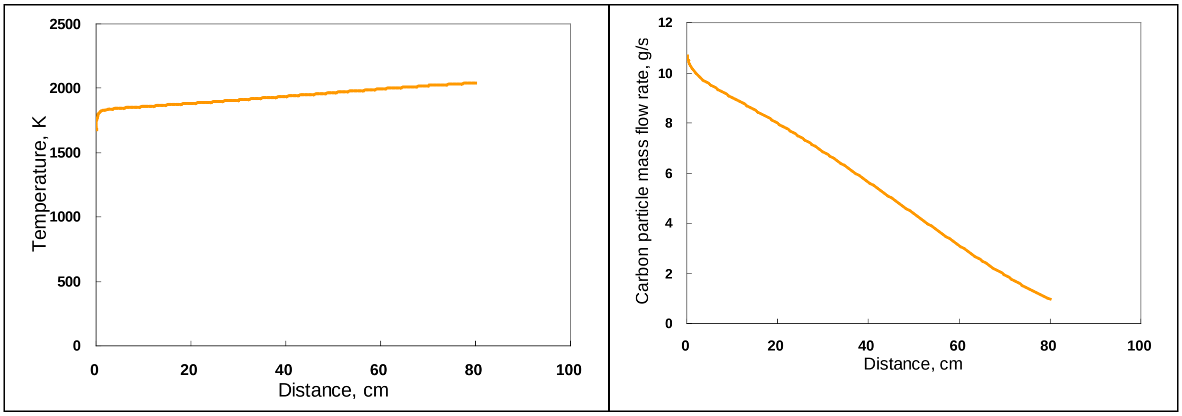


Figure 3. Results from the second PFR

Simulations were also carried out investigating the effect of oxygen concentration in the enriched secondary combustion air on the carbon particles combustion. In these simulations the oxygen content in the secondary combustion air was increased from 23% (the value used in the simulations above) to 35%. Increasing the oxygen content in the enriched air significantly impacts the combustion of the carbon particles in the 2nd PFR. As Fig. 4 below indicates, the conversion in the reactor reaches 91%, which brings the total conversion of carbon particles in the three-reactor cascade (simulating the water-cooled cyclone chamber) to 96%.

In summary, the simulations presented here indicate that complete conversion of the gaseous components and substantial conversion of the carbon particles found in the off-gas is expected in the water-cooled cyclone chamber. As noted above, and also discussed in the previous Quarterly Reports, extensive search of the technical literature failed to generate any information that is available about the global kinetics of carbon combustion under conditions that are likely to be encountered in the water-cooled cyclone chamber that is being simulated here; therefore, the results presented here should be validated experimentally.



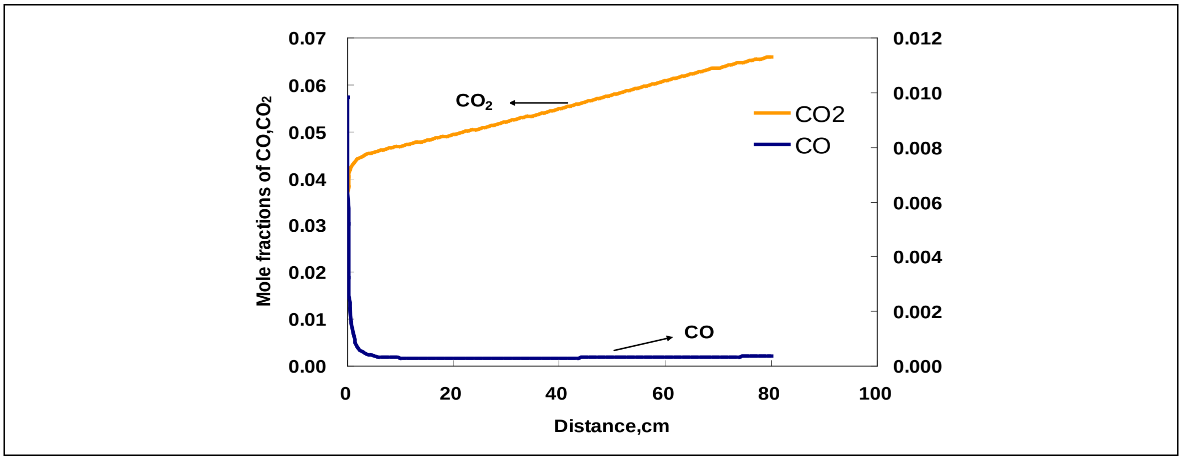


Figure 4. Impact of oxygen content [Results from the second PFR]

For example, prior simulations (presented in the previous reports) indicated the great sensitivity of the carbon conversion to the activation energies of Reactions (1) and (2). Unfortunately, we have been unable to locate any reliable literature data for the activation of Reaction (3), which as a result was taken from [2].

4. References:

[1] H. S. Fogler, "Elements of Chemical Reaction Engineering," Prentice-Hall, Int. Series, 4th Edition, 2006.

[2] L. Jelemensky, J. Markos, R. Zajdlik and B. Remiarova. Modelling of Nonlinear Behaviour During Combustion of Single Coal Char Particle. 27th International Conference of the Slovak Society of Chemical Engineering, Tatranske Matliare, May 22-26, 2000.

[3] B. Beshty. A Mathematical Model for the Combustion of a Porous Carbon Particle. Combustion and Flame 32, 295-311, 1978.

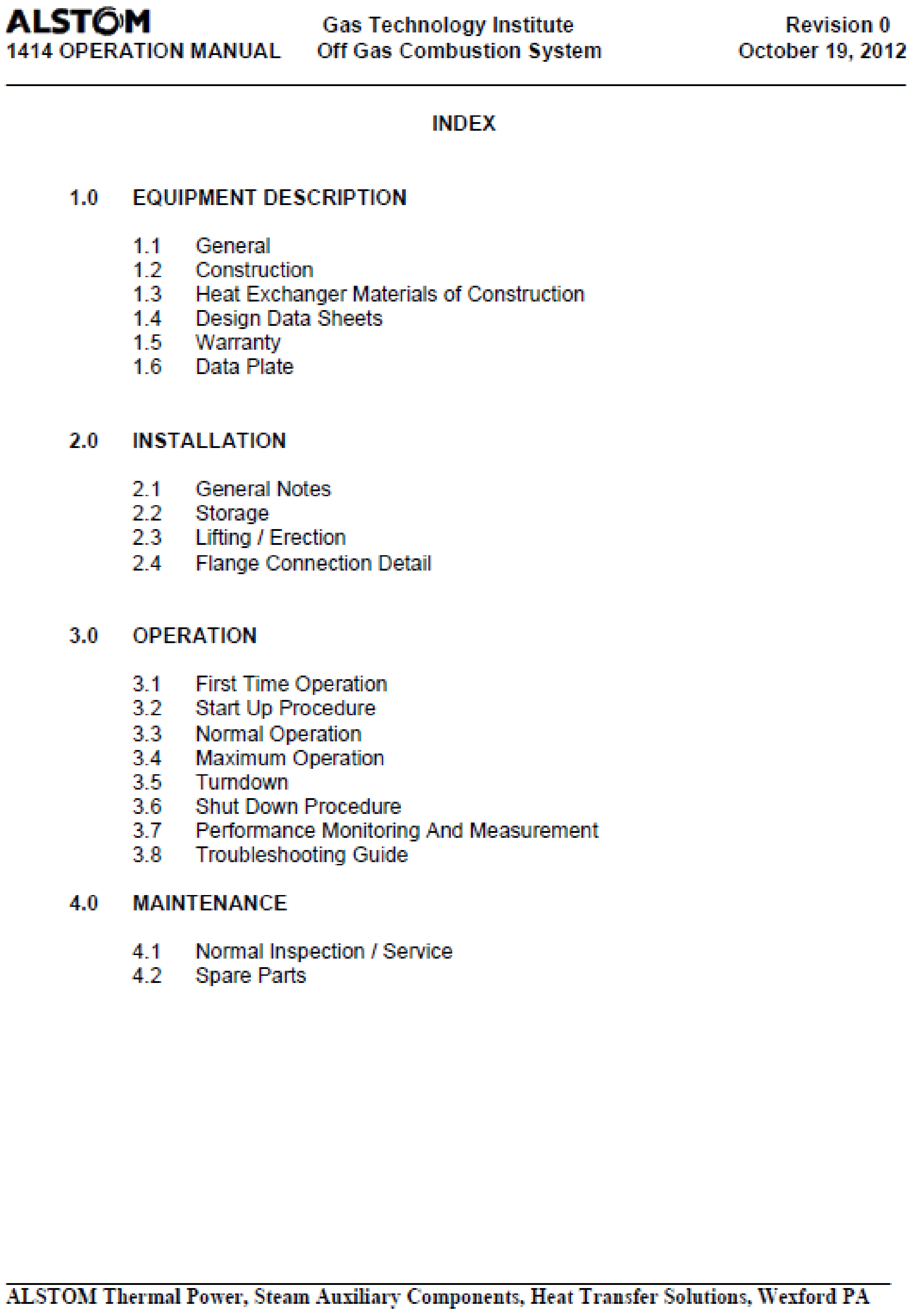
[4] P. A. Tesner. The Activation Energy of Gas Reactions with Solid Carbon. 8th international symposium on combustion, California Institute of Technology, Pasadena, California, August 28-September 2, 1960.

[5] http://www.me.berkeley.edu/gri_mech/

[6] http://garfield.chem.elte.hu/Combustion/sox.htm

APPENDIX D

O&M Manual Index



ALSTOM
1414 OPERATION MANUAL

Gas Technology Institute
Off Gas Combustion System

Revision 0
October 19, 2012

INDEX

ALSTOM Thermal Power, Steam Auxiliary Components, Heat Transfer Solutions, Wexford PA

INDEX (Continued)

APPENDIX E

Solex High Temperature Cooler Thermal Design

[Summary of test results under subcontract S124]

Solex Thermal Science Inc.
100, 3595 - 114 Ave SE, Calgary, AB, Canada T2Z 3X2
p. (403) 254-3500
f. (403) 254-3501
www.solexthermal.com

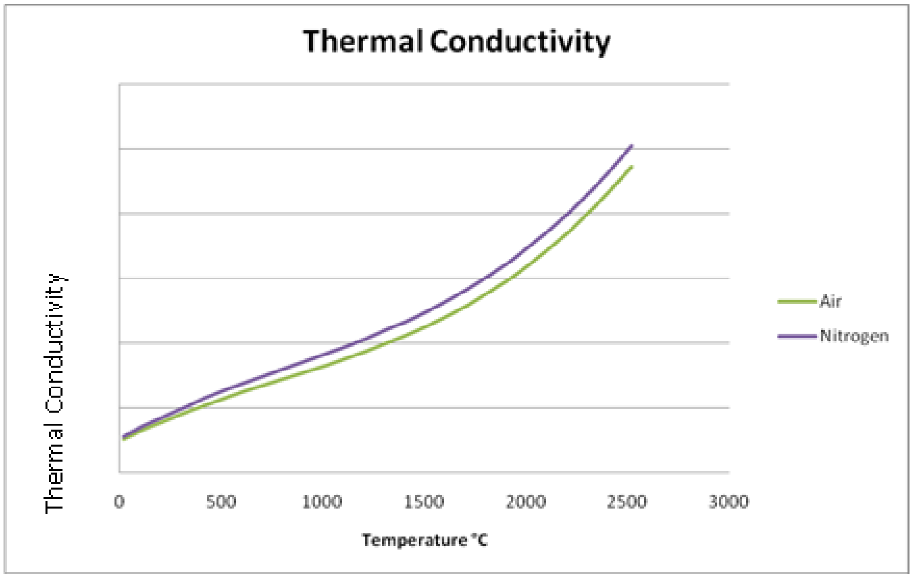
Solex thermal science has been designing and building heat exchangers for cooling bulk solids for the last couple of decades. The highest operating temperature of these units has been in the 400°C to 500°C range. Due to the demand for high temperature coolers and for energy recovery, Solex looked more closely at their design and eliminated the problem areas that could not handle bulk solid temperatures over 500°C. Finite element modeling was done to optimize the design of the heat exchanger plates and a pilot test unit was built. This report outlines the results of the first pilot test run done with this test unit at Superior Graphite with product temperatures up to 2000°C.

2.0 SUMMARY:

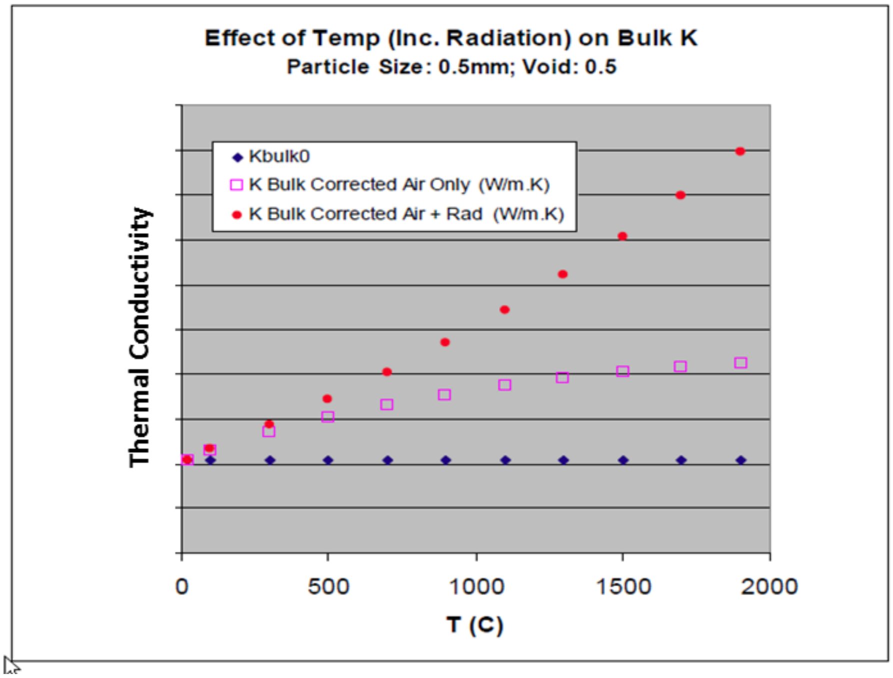
Cooling performance of the test unit was much better than expected
Casing remains cool to the touch during testing except at very top of top bank
Plates looked like new when testing was completed.

3.0 OBSERVATIONS AND DISCUSSION:

Original thermal models were run using an average thermal conductivity of the product measured at ambient conditions. Since the thermal conductivity of the air or nitrogen in the void space of the particles increases significantly with temperature, the thermal conductivity of the bulk solid also increases significantly with temperature, see chart below.



This alone does not explain the increase in performance, so we realized that the radiation component of heat transfer was much larger than we expected. This component of heat transfer is being added to our modelling software. The effect of radiation can be seen in the plot below.



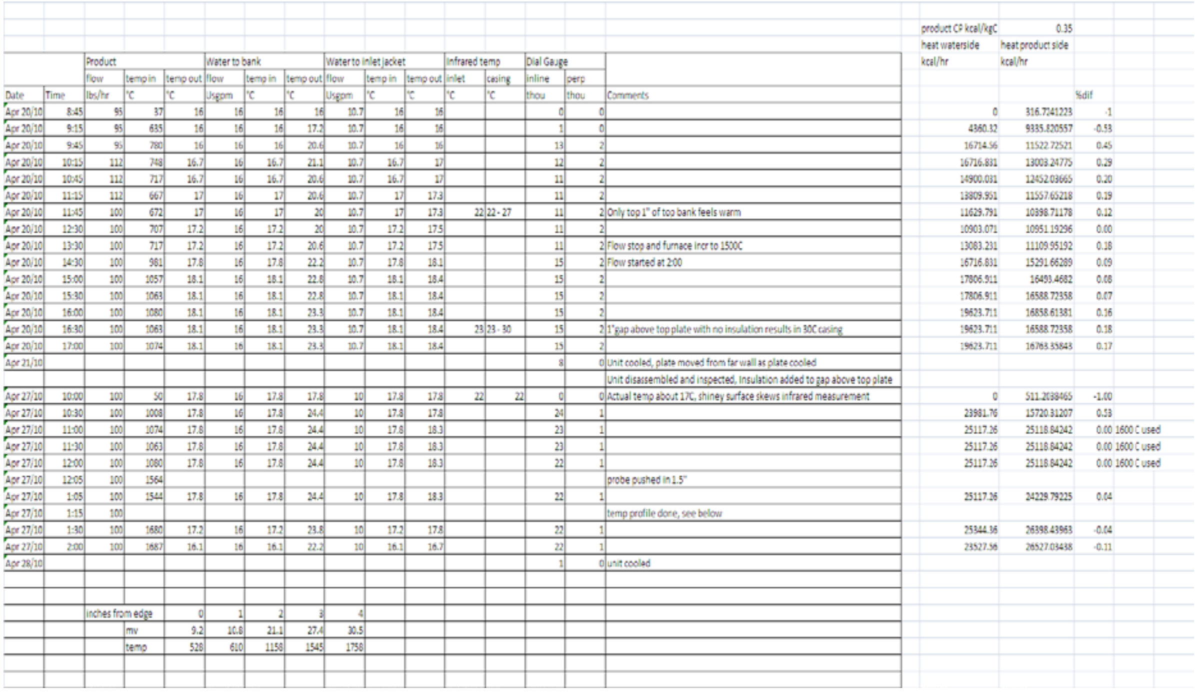
Since the casing remained cool to the touch, high temperature stainless is not required. A bolted construction which allows for thermal expansion is also not required since the casing remains at near ambient conditions. There was a small area between the top of the top plate and the bottom of the graphite liner in the inlet hopper that was not insulated and the hot product was able to run up against the casing. Since the flow was stagnant only a small amount of heat conducted through the product to the casing wall. This area was insulated in between runs and the final run at 2000°C temp at the furnace showed no significant heating at this location.

The plates are fairly close together in the unit, therefore larger chunks of material that may sluff off the walls of the furnace can build up on top of the plates. For this reason the unit needs to be easily dropped or accessed for cleaning. There is also a need to ensure that the furnace is absolutely clean between runs to ensure there is no cross contamination.

The center plate of the top bank which was exposed to the highest product temperature has been sent back to Solex for analysis.

Results from pilot test will be compared to finite element results to validate modelling.

4.0 DATA:



																product CP kcal/kgC	0.35		
		Product			Water to bank			Water to inlet jacket			Infrared temp		Dial Gauge			heat waterside	heat product side		
		flow	temp in	temp out	flow	temp in	temp out	flow	temp in	temp out	inlet	casing	inline	perp		kcal/hr	kcal/hr		
Date	Time	lbs/hr	°C	°C	Usgpm	°C	°C	Usgpm	°C	°C	°C	°C	thou	thou	Comments			%dif	
Apr 20/10	8:45	95	37	16	16	16	16	10.7	16	16			0	0		0	316.7241223	-1	
Apr 20/10	9:15	95	635	16	16	16	17.2	10.7	16	16			1	0		4360.32	9335.820557	-0.53	
Apr 20/10	9:45	95	780	16	16	16	20.6	10.7	16	16			13	2		16714.56	11522.72521	0.45	
Apr 20/10	10:15	112	748	16.7	16	16.7	21.1	10.7	16.7	17			12	2		16716.831	13003.24775	0.29	
Apr 20/10	10:45	112	717	16.7	16	16.7	20.6	10.7	16.7	17			11	2		14900.031	12452.03665	0.20	
Apr 20/10	11:15	112	667	17	16	17	20.6	10.7	17	17.3			11	2		13809.951	11557.65218	0.19	
Apr 20/10	11:45	100	672	17	16	17	20	10.7	17	17.3	22	22 - 27	11	2	Only top 1" of top bank feels warm	11629.791	10398.71178	0.12	
Apr 20/10	12:30	100	707	17.2	16	17.2	20	10.7	17.2	17.5			11	2		10903.071	10951.19296	0.00	
Apr 20/10	13:30	100	717	17.2	16	17.2	20.6	10.7	17.2	17.5			11	2	Flow stop and furnace incr to 1500C	13083.231	11109.95192	0.18	
Apr 20/10	14:30	100	981	17.8	16	17.8	22.2	10.7	17.8	18.1			15	2	Flow started at 2:00	16716.831	15291.66289	0.09	
Apr 20/10	15:00	100	1057	18.1	16	18.1	22.8	10.7	18.1	18.4			15	2		17806.911	16493.4682	0.08	
Apr 20/10	15:30	100	1063	18.1	16	18.1	22.8	10.7	18.1	18.4			15	2		17806.911	16588.72358	0.07	
Apr 20/10	16:00	100	1080	18.1	16	18.1	23.3	10.7	18.1	18.4			15	2		19623.711	16858.61381	0.16	
Apr 20/10	16:30	100	1063	18.1	16	18.1	23.3	10.7	18.1	18.4	23	23 - 30	15	2	1"gap above top plate with no insulation results in 30C casing	19623.711	16588.72358	0.18	
Apr 20/10	17:00	100	1074	18.1	16	18.1	23.3	10.7	18.1	18.4			15	2		19623.711	16763.35843	0.17	
Apr 21/10													8	0	Unit cooled, plate moved from far wall as plate cooled				
															Unit disassembled and inspected, Insulation added to gap above top plate				
Apr 27/10	10:00	100	50	17.8	16	17.8	17.8	10	17.8	17.8	22	22	0	0	Actual temp about 17C, shiney surface skews infrared measurement	0	511.2038465	-1.00	
Apr 27/10	10:30	100	1008	17.8	16	17.8	24.4	10	17.8	17.8			24	1		23981.76	15720.31207	0.53	
Apr 27/10	11:00	100	1074	17.8	16	17.8	24.4	10	17.8	18.3			23	1		25117.26	25118.84242	0.00	1600 C used
Apr 27/10	11:30	100	1063	17.8	16	17.8	24.4	10	17.8	18.3			23	1		25117.26	25118.84242	0.00	1600 C used
Apr 27/10	12:00	100	1080	17.8	16	17.8	24.4	10	17.8	18.3			22	1		25117.26	25118.84242	0.00	1600 C used
Apr 27/10	12:05	100	1564												probe pushed in 1.5"				
Apr 27/10	1:05	100	1544	17.8	16	17.8	24.4	10	17.8	18.3			22	1		25117.26	24229.79225	0.04	
Apr 27/10	1:15	100													temp profile done, see below				
Apr 27/10	1:30	100	1680	17.2	16	17.2	23.8	10	17.2	17.8			22	1		25344.36	26398.43963	-0.04	
Apr 27/10	2:00	100	1687	16.1	16	16.1	22.2	10	16.1	16.7			22	1		23527.56	26527.03438	-0.11	
Apr 28/10													1	0	unit cooled				
		inches from edge		0	1	2	3	4											
			mv	9.2	10.8	21.1	27.4	30.5											
			temp	528	610	1158	1545	1758											

APPENDIX F

Selected Excerpts from SGC Progress Reports on ACHP Installation and Shakedown

December 13, 2012

Major Progress Summary:

- Water tank installed, pump and indoor piping are ready.
- Pilot burner parts received.
- Water cooled flanges are fabricated and refractory work finished.
- Afterburner is assembled.
- Control Room built.

Progress:

1. Installation of water tank, pump and piping in R&D area: A water tank for storage of cooling water has been made and placed in R&D area. Water pump and power control has been installed, indoor cooling water section is almost ready now.
2. Pilot burner parts received on December 6, 2012
   Package includes ignition transformer and ignition cable, 3” nozzle mix gas pilot, ½” std regulator, ½” limiting orifice valve and 3” butterfly valve.
3. Water cooled flanges were fabricated and refractory work is finished. Afterburner was assembled and installed


01/30/2012 12:16

Bottom (Chamber 3, 4, 5, 6)


01/30/2012 12:15

Top (chamber 2 and 3)

4. Control Room built for data acquisition:


12/12/2012

Chamber 1 and cross section

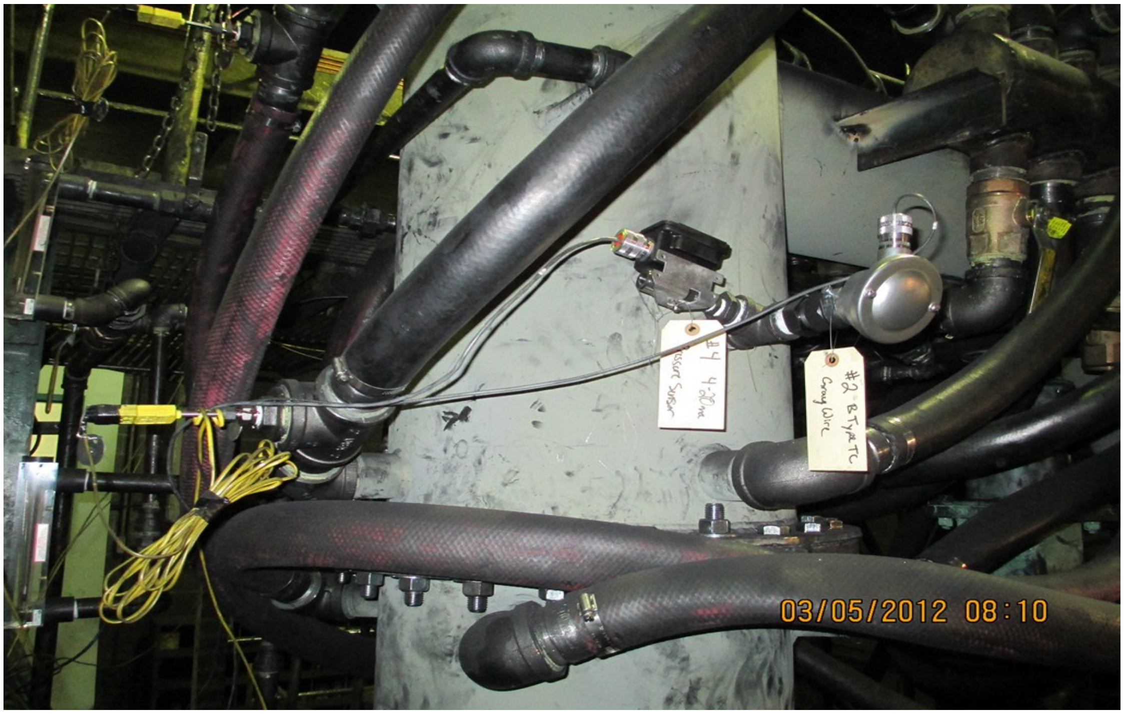
<u>March 6, 2013</u>

Major Progress Summary:

- Internal cooling water circle done, leak test finished
- Gas train for pilot burner assembled
- All sensors by SG installed
- Green coke material for testing prepared (wood river green shot coke prescreened and dried, 2.5 tons for hot trail)
- Installation of gas train→ will be done today/tomorrow
- Connection of outside cooling water→will be done by next week
- Calibration gas for GC→order placed, deliver by March 22

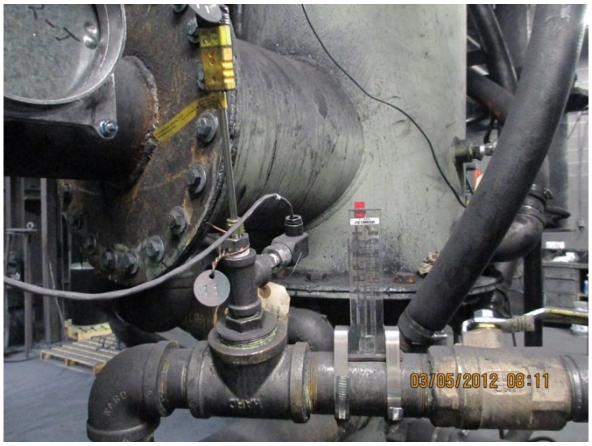
Progress:

All internal circles cooling water pipe for GTI afterburner installed; sensors and data acquisition system installed.


#4 4-20ma
Pressure Sensor
#2 B Type TC
Gray Wire
03/05/2012 08:10

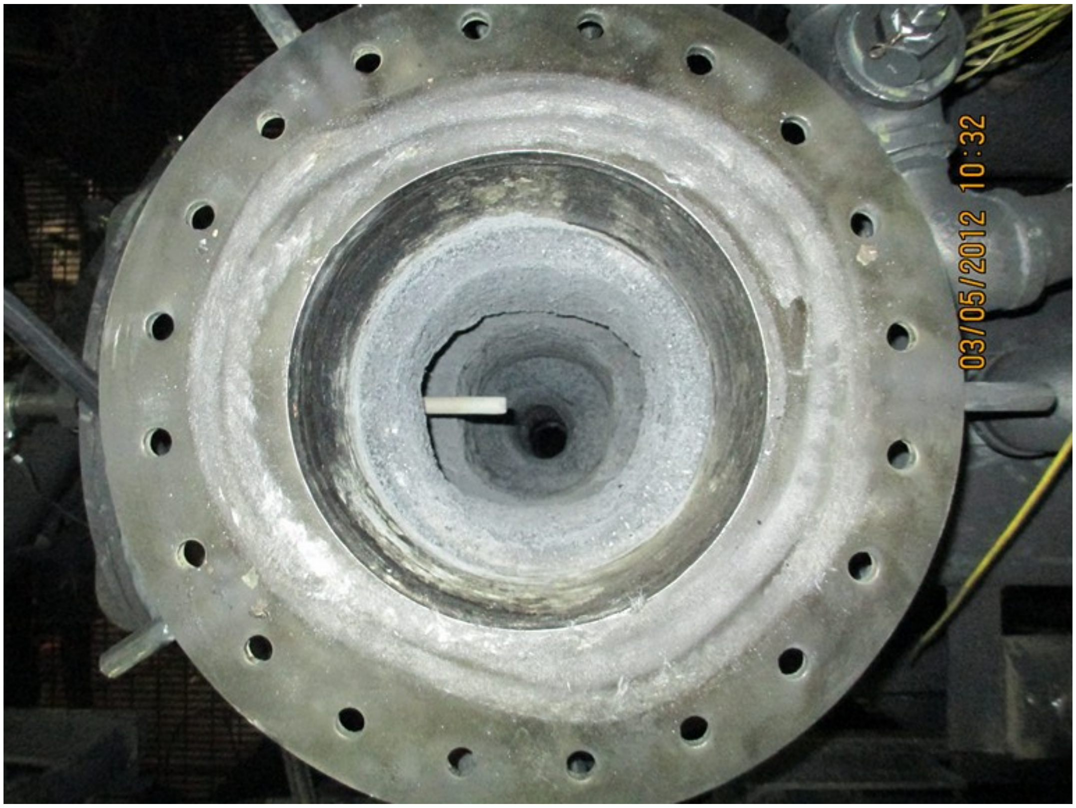

03/05/2012 0


03/05/2012 08:10


03/05/2012 08:11


03/05/2012 08:08

Rodding flue use current system:



Thermocouple not in the way of rodding

Safety Analysis

Furnace Operations:

1. Interface between furnace flue and afterburner has to be kept tight to avoid uncontrolled early combustion. Use refractory rope to seal the gap between furnace flue exit and afterburner inlet.
2. Afterburner Tee top cap need to be modified for easy removal, watch out for early combustion, furnace operator needs to be very cautious when put cap back.
3. Exhaust gas pipe temperature needs to be closely monitored. Use dilute air to bring down the exhaust gas temperature, or add cooling water jacket, set up hand rail around the exhaust gas pipe for precaution and warning sign of hot pipe.
4. Nature gas line safety. Use certified contractor to assemble the gas train, or GTI provide the assembled gas train directly.
5. Pilot burner always on. The control of pilot burner need to be double

checked to guarantee the natural gas shut down when there are no flames. Audible alarm can be installed for warning.

6. Power failure and lose cooling water. Start force cooling mode.
7. Tornado event. Turn off power input and keep cooling water running to cool down furnace. Evacuate to tornado shelter.
8. First time operation of afterburner, to protect the refractory layer in afterburner, increase furnace temperature as slow as possible. Bag house failure. Stop the trial and cool down furnace slowly, use ventilation fan in R&D area to maintain good ventilation.
9. Possible accumulation of H_2 on roof. Maintain good ventilation around pilot furnace area or install ventilation fan on roof.
10. Cooling water temperature should be maintained to prevent steaming.

Safety Procedure for GTI afterburner testing

Cold trial:

Before the system started, check DAQ system and sensors; check cooling water flow and afterburner system pressure; Open sliding valve at the bottom of the afterburner and check pressure again to see the dilute air can be sucked into the system.

Before hot trial, a toxic gas detector will be installed to monitor the combustible gas content in the R&D area.

Hot trial:

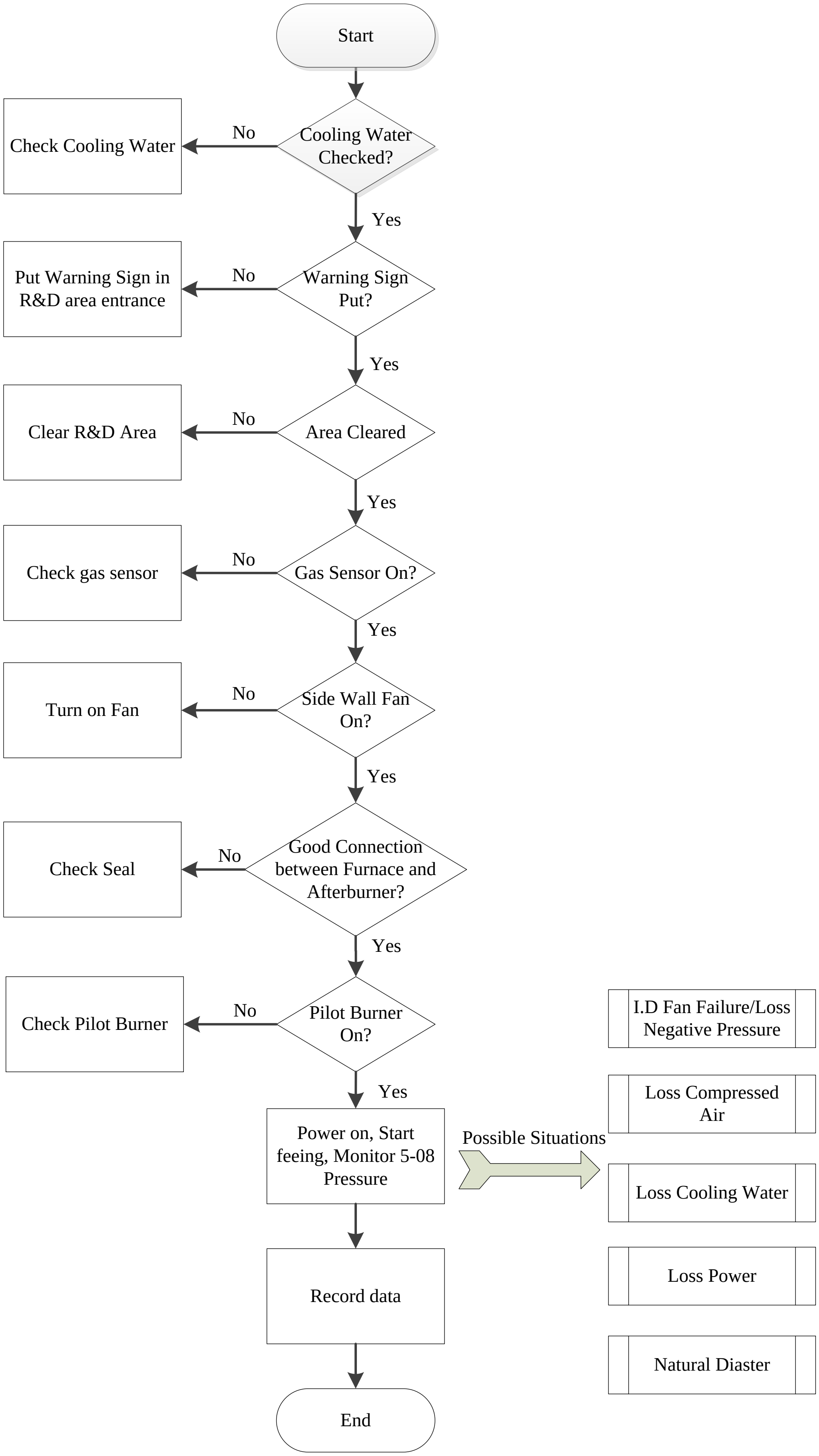
1. Clear the smoke zone outside R&D, no smoking allowed near the R&D area, place proper warning sign.
2. Check DAQ system and sensors including the off gas sampler. Make sure afterburner is working properly, closely monitoring cooling water flow and pilot flame.
3. Start feeding furnace with green coke and discharge at a rate of 25 lbs/hr, introduce flue gas into the afterburner and ignite the flue gas by gas pilot to see if a stable flame is established. If not, increase throughput to 50 lbs/hr and try again. If still not, then increase the furnace temperature, see if we get

stable flame. Once a stable flame is detected, collect data.

4. After collected enough data, increase throughput slowly, closely monitor system temperatures, and increase temperature and record data. If the system is working well and stable, try increase throughput to 100 lbs/hr.

Safety Operation of furnace:

1. All cooling water lines need to be checked before the furnace run, not only for the pilot furnace, but also for the afterburner.
2. Proper warning signs need to be place on the entrances to R&D area. Smoke zone outside R&D area need to be cleared during the trial.
3. Toxic gas sensor needs to be placed in R&D area with alarm tested.
4. Side wall fan needs to be on all the time during the trial, make sure good ventilation air flow in the R&D area.
5. All unrelated personnel should not enter the R&D area during the trial. No forklift should be parked near the R&D area.
6. Interface between furnace flue and afterburner has to be kept tight to avoid uncontrolled early combustion. Use refractory rope and/or high temperature insulation blanket to seal the gap between furnace flue exit and afterburner inlet.
7. Exhaust gas pipe temperature needs to be closely monitored. Use dilute air to bring down the exhaust gas temperature.
8. Safety prevention chains need to be installed on the 3rd floor in the R&D area, including the top of the furnace and area open to the pilot burner, to prevent operators from falling off the platform.
9. Pilot burner should be on all the time during the trial. Furnace operator needs to pay attention to warning signal or alarms from the burner primary control system.
10. When losing compressed air, turn off pilot burner immediately, close all the valves on the gas line to avoid sudden back up of gases.
11. When there is power outage or tornado event caused power outage, stop the feeding control and turn off the pilot burner, switch to force cooling mode.
12. Current evacuation plans and emergency shutdown plans will stay in effect in case of uncontrolled situation happening.


Start
Cooling Water Checked?
No
Check Cooling Water
Yes
Warning Sign Put?
No
Put Warning Sign in R&D area entrance
Yes
Area Cleared
No
Clear R&D Area
Yes
Gas Sensor On?
No
Check gas sensor
Yes
Side Wall Fan On?
No
Turn on Fan
Yes
Good Connection between Furnace and Afterburner?
No
Check Seal
Yes
Pilot Burner On?
No
Check Pilot Burner
Yes
Power on, Start feeing, Monitor 5-08 Pressure
Possible Situations
I.D Fan Failure/Loss Negative Pressure
Loss Compressed Air
Loss Cooling Water
Loss Power
Natural Diaster
Record data
End

March 15, 2013

Off-Gas Combustion System Test Procedures

This document provides an overview of the test procedures for off-gas combustion system at Superior Graphite Research Facility. The test procedures have been established to insure safety and data quality.

Prior to start and operate the off-gas combustion system, combustion air flow rates (both main and pilot) should be checked and adjusted to nominal values. Set required air pressure in the off-gas combustion chamber by adjusting speed of the exhaust gas fan. Both primary air and secondary air are introduced into the system. The air flow rates should be controlled by air flow meters installed at each air line.

Startup procedure of the combustion system is very important to insure safety. Gas pilot should be ignited first before the off-gas is introduced into the off-gas combustion system. The gas pilot should be operated at lean conditions (10-50% excess air). Flame detector (sensor) should indicate that the gas pilot is in operation and there is a pilot flame in the off-gas combustion chamber. If flame sensor does not detect the flame this means that there is no pilot flame in combustion chamber or the flame sensor does not see the flame. Shut off natural gas to the gas pilot and check if natural gas and air are supplied to the pilot burner, spark ignition system works properly or the flame is visible from the sensor location.

When the pilot flame is established, the off-gas can be introduced in the combustion chamber. Observe thermocouple's reading (temperature rise in the combustion chamber in the beginning of the process) to be sure that the off-gas is combusted. If the off-gas combustion process is stable then the gas pilot can be shut off. **(We will keep it on all the time)** The flame sensor will indicate that there is a flame in the off-gas combustion chamber. When the main flame is off, the gas pilot should be started right away to ignite the off-gas. If the off-gas can't be ignited (flame sensor doesn't detect the flame) then the off-gas supply should be stopped**. (We leave it on but increase air flow in combustion chambers to purge the system)**

When the main flame in the off-gas combustion chamber is established and the system operates properly, negative gas pressure in the combustion chamber and excess air should be controlled to keep nominal values of the parameters. The value of gas pressure is dictated by the process and controlled by the exhaust gas fan**. (Damper will be added)** Excess air is 10-30% and can be changed by restricting the main air flow rate. Gas analyzer at the exhaust gas should be used to control the excess air by measuring oxygen content (2-5%, dry basis) in the exhaust gas.

The following parameters should be measured and/or monitored in each test in order to control the combustion process:

- Flame presence in the off-gas combustion chamber
- Negative pressure in module 1 of the off-gas combustion chamber
- Off-gas composition
- Exhaust gas composition
- Water inlet and outlet temperatures for each module, chock ring, and lid

- Off-gas temperature
- Gas temperature in modules 1, 3, 5, and 6
- Gas pressure in modules 2, 4, and 6
- Water total flow rate
- Primary air flow rate
- Secondary air flow rate

Many parameters will be measured and monitored. Three data acquisition systems and three computers will be used to measure and calculate (1) temperatures, pressures, and flow rates; (2) off-gas composition; and (3) exhaust gas composition. Additional parameters will be added as necessary to control and monitor the performance of the off-gas combustion system.

Cold Trial:

1. **Afterburner gas lines tested.**

   Pilot burner:
   Compressed air line pressure setting 3 oz/in^2.
   Natural gas line pressure setting at 1 psi.
   Ratio regulator (in red circle) adjusted to correct position.
   Wiring for ignition and flame safe control connected.

2. **Water cooled off gas sampler (in red circle) installed.**

3. **Gas tubing for taking gas sample installed.**

03/13/2012 06:39

4. **Data acquisition system installed.**

03/13/2012 06:35

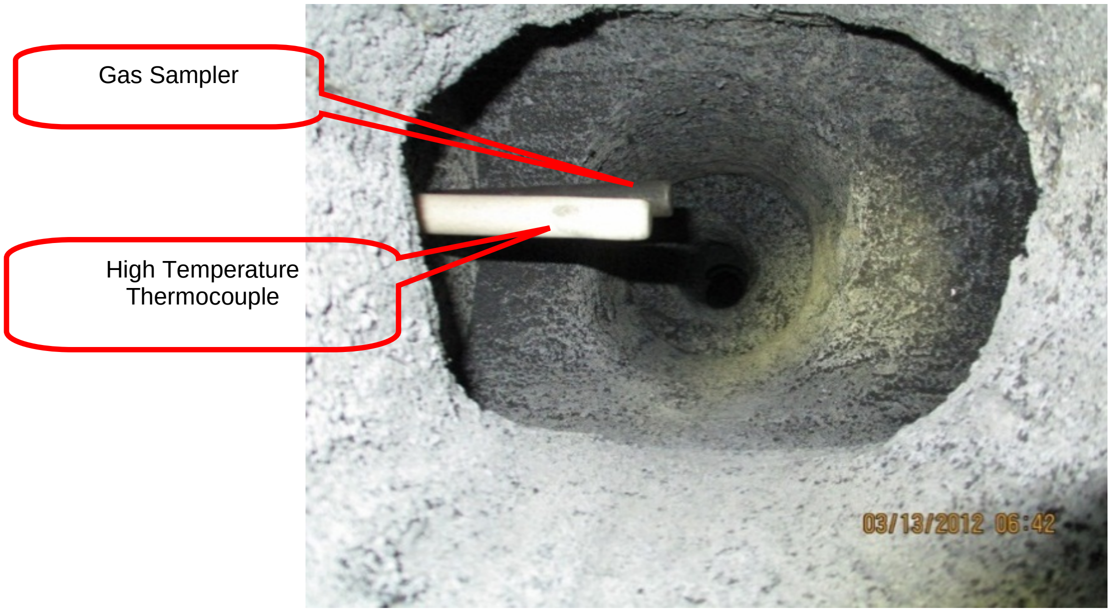
03/13/2012 06:37

5. **Cooling water flow tested:** For the two cooling water circle, each circle is around 260 GPM, total flow of 520 GPM, satisfy the design of 505 GPM.

03/13/2012 06:47

03/13/2012 06:48

6. Furnace cold run



The current off gas system has enough pulling power, and the slightly negative pressure can be adjusted by adjusting damper, or the compress air flow rate to the module 1 and 2.

April 2, 2013

Summary:

- All afterburner parts including pilot burner installed and tested.
- GC and gas analyzer ready.

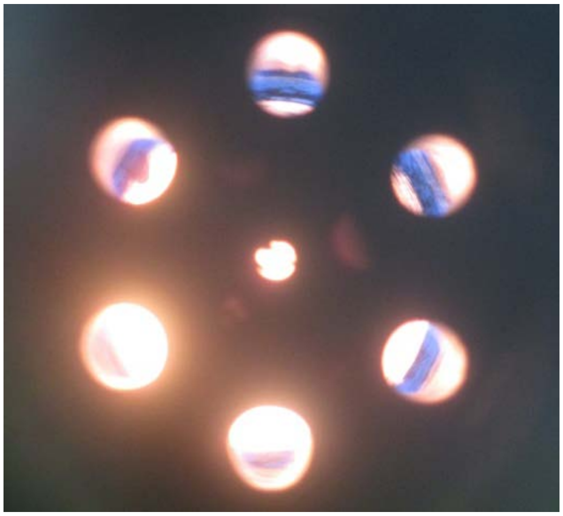
Background: The purpose of this pilot furnace trial is for concept proof of the GTI afterburner and obtain data for energy balance calculations. The plan is to warm up furnace first then introduce green coke, when the volatiles from the furnace reach combustion chamber of the afterburner, a controlled combustion will take place and recover heat from the flue gas.

As part of the preparation, on March 28 and 29 (Thursday and Friday), Hop R&D team starts to tune up the refractory layer in the afterburner under furnace operation condition, after that the furnace was let cool during the weekend as usual. On Apr 1 GTI engineers start to set up GC and gas analyzer, tune up the pilot burner and calibrate the thermocouples. System is ready for hot trial on Tuesday. Per GTI's suggestion, the afterburner will be maintained at slightly negative pressure so that the furnace flue gas can be suck through the swirling channel to the combustion chamber.

June 19, 2013

Observations during/after the hot trial:

- The whole system was running very stable at low throughput at 22 lbs/hr, though the flue gas shows extra Oxygen in it (~13%). After increase throughput, the pilot burner became unstable, and it is hard to maintain the burner on at current burner settings. Below shows the flame image when running only on pilot burner and without pilot burner.



(a) Combustion on Pilot burner only

(b) Combustion without pilot burner

The configuration of the pilot burner and the setting of gas train is shown below:

(a) Pilot Burner

(b) Compressed Air

(c) Natural gas after pressure regulator

- Pressure sensor in Tee section of afterburner was very helpful to monitor the furnace off gas pressure, so to avoid damage to pilot furnace. The sensor is shown in red circle below.

- During the trial, water (red circle below) dropping from the bottom cone of the afterburner, we believe it is mainly the condensation water (pH=4.6). After the trial we opened the section, mud like material is found filling the bottom cone. The solids are deposit from previous curing refractory and this hot trial.

(a) Bottom cone of the afterburner collecting water

(b) Water dripping down and the mud like material in the cone section

(c) Material coming out of the cone section

- No metal deposition was found in the furnace flue tube. During the whole trial no rodding is needed. Carbon deposit was found in the afterburner combustion chamber gaps. The combustion of carbon material is not complete according to the findings

(a) Gap between the chamber 1 and 2, decent amount of carbon material was filling the gas

- The thermocouple in combustion chamber 1 is not showing correct numbers. It was not correctly calibrated for B type thermocouple. All the other thermocouple is working properly.

- The cooling water temperature only increased about 1~1.5 °C after passing through the channels during the trial.

- The volatile in the green coke is 10.10%, and dropped to 0.24% after calcination at 900 °C. Ash content in the green coke dropped from 0.32% to 0.13%. Sulfur dropped from 5.90% to 1.64%.

Summary:

- Successfully created stable controlled combustion in the afterburner for throughput at 22 lbs/hr. System pressure and temperature stabilized with pilot burner always on, stable combustion was observed in the combustion chamber. First time ever proved the concept of controlled combustion of volatiles from green coke calcination.
- System became unstable at throughput of 35 lbs/hr, pilot burner went off after 45 mins. After the pilot burner went off, the combustion self-sustained for about 10 mins and went off after the system temperature drops. Pilot burner settings need to be fine-tuned.
- System failed at throughput of 50 lbs/hr, pilot burner and combustion went off right away after increased the throughput. Increasing throughput does not help solving the problems.
- GTI afterburner maintained good shape, no damage was found after the trial.
- A pile of dusts were found filling the bottom cone of the afterburner, soaked with slightly acidic condensation water and forming mud like mixture.
- The volatile in the green coke is 10.10%, and dropped to 0.24% after calcination at 900 °C. Ash content in the green coke dropped from 0.32% to 0.13%. Sulfur dropped from 5.90% to 1.64%.

Conclusions:

- First time successfully run the GTI afterburner. Proved the concept of volatile combustion for energy recovery.
- The green coke was well calcined during the low throughput test.
- Carbon material accumulated inside the afterburner. According to original design, all the carbon material should be combusted.
- Significant condensation water forming during the short trial.
- The next GTI hot trial will be scheduled to the week of July 8.

Action Items:

1. GTI run the gas analysis for the flue gas sample.
2. GTI calibrate the thermocouple (B type TC) at combustion chamber 1.
3. GTI work on tuning the pilot burner condition to make it stable under various conditions.
4. GTI check the amount of compressed air needed in the combustion chambers especially at higher throughput.
5. GTI check the data obtained during this hot trial and decide if there are other data needed.
6. GTI bring the gas analyzer and the GC for next hot trial.
7. HTT R&D cleans the flow meter before next hot trial.

July 9-11, 2013

Summary:

- Successfully maintained combustion condition in the afterburner for throughput up to 100 lbs/hr.
- Pilot burner was kept on during the trial in order to maintain stable combustion. Changes were made to guarantee pilot flame on.
- Furnace flue GC results show increased Hydrogen percentage as the throughput increased from 22 lbs/hr to 100 lbs/hr.
- GTI afterburner maintained good shape, no damage was found after the trial. Pilot furnace conical plate and discharge tube were found slightly damaged after open the furnace after the run.
- The volatile drops from more than 10% to less than 0.5%. Sulfur level drops from 6% down to less than 2%, compare to traditional green coke calcination which usually still maintains 85% of sulfur level after calcination.
- Safety procedure was modified to prevent potential accident/hazards when running green coke.

Background:

The purpose of this pilot furnace trial is to improve the GTI afterburner after the second hot trial which already proved the feasibility of combusting volatile during green coke calcination. In addition to reproduce previous 22 lbs/hr throughput in hot trial 2, this trial targeting on running at higher temperature (may go up to 1500 °C) and higher throughput (may up to 100 lbs/hr).

Observations:

- Safety precaution: Clear warning signs were posted at every R&D entrance; toxic gas sensor (detect O_2, H_2S, CO and combustible gas) was placed on top of furnace and afterburner.

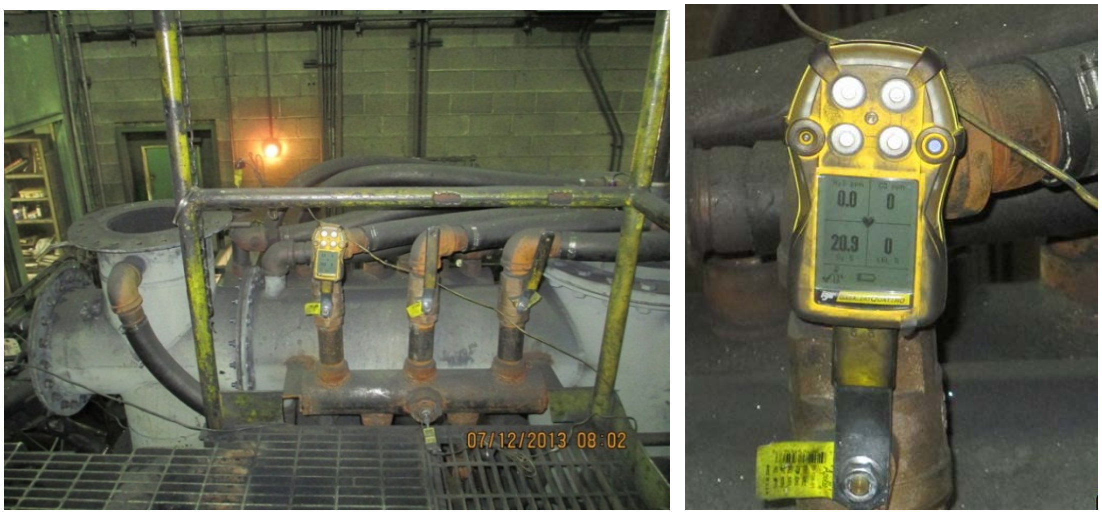


Fig 1. Gas detector for working area safety

- On July 9, the GC and gas analyzer was connected to the system. GC was preheated before the trial. The gas cylinder for the gas analyzer brought by GTI was connected and all measurement points are tested.

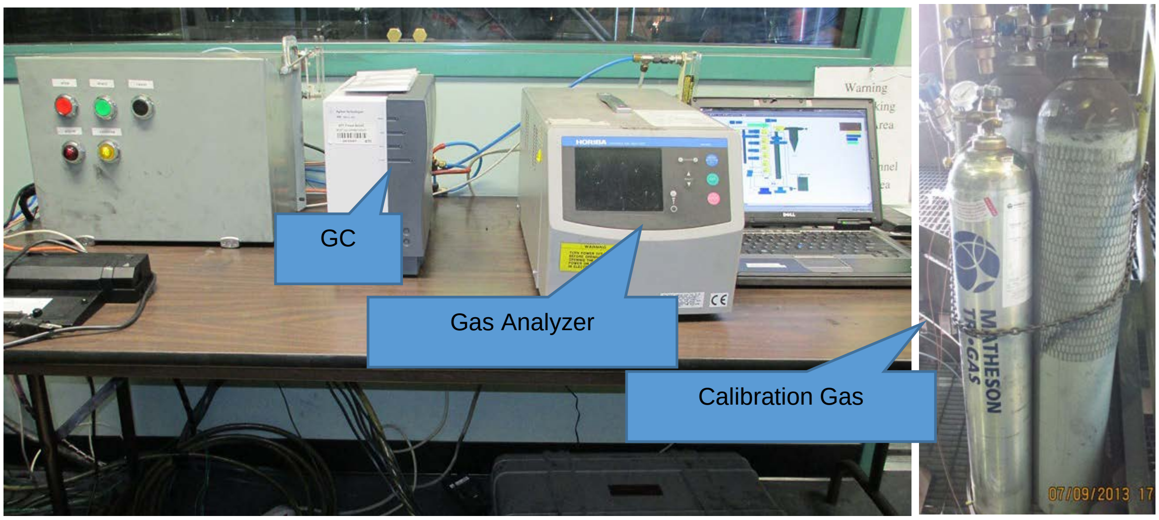


Fig 2. GC for flue gas composition analysis and gas analyzer for off gas analysis

- Adjustment was made to the pilot burner to get a stable/better pilot flame. The pressure regulator of the gas train compressed air was connected to the combustion chamber, so it can feel the pressure change in the chamber (the white tube).

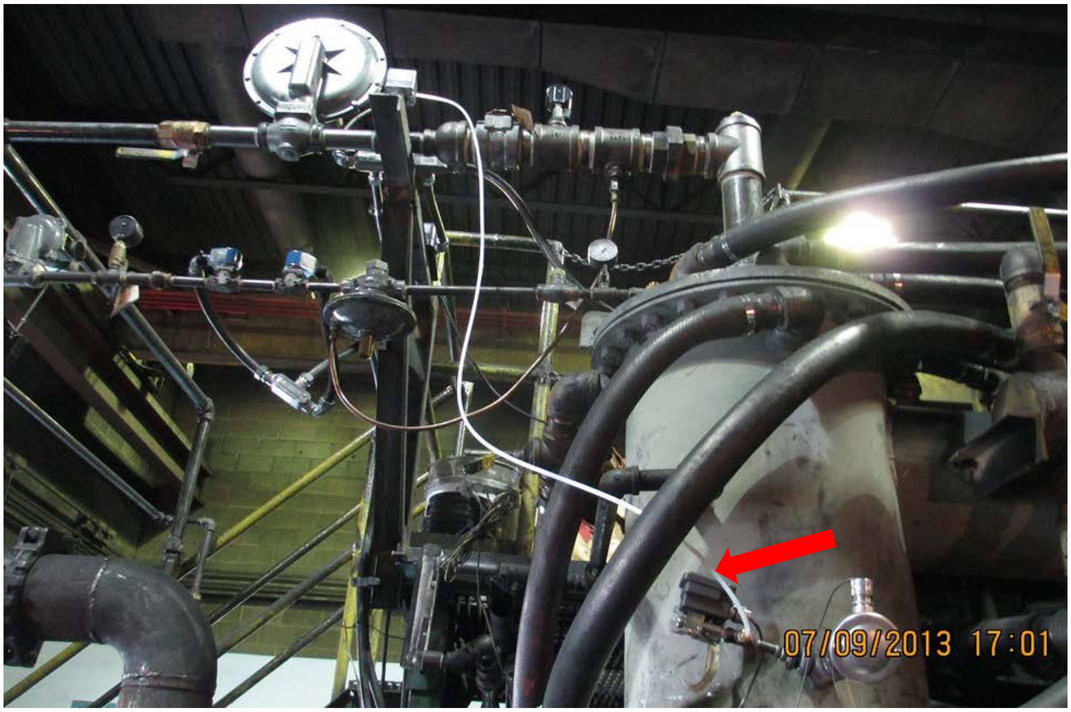
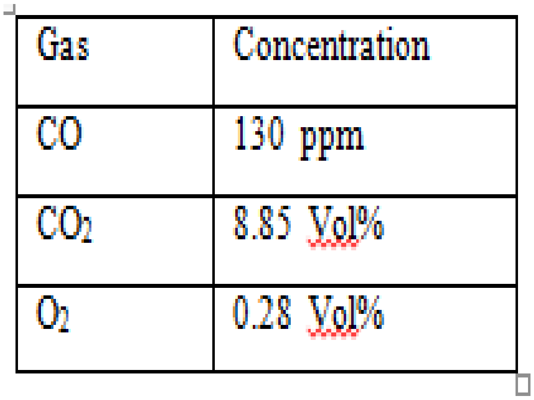


Fig 3. The compressed air pressure regulator connected to combustion chamber

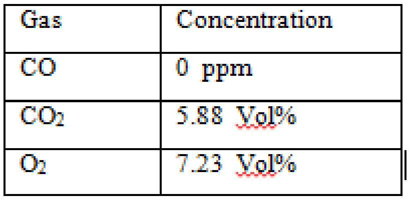
- On July 10, start from 9:40 AM, Stable flame was observed and Gas analyzer shows composition:



Gas	Concentration
CO	130 ppm
CO_2	8.85 Vol%
O_2	0.28 Vol%

Fig 4. Pilot flame (no volatile, no extra compressed air)

At 10 AM, start to add extra compressed air, combustion chamber (CC) 1= 5SCFM, CC2=1 SCFM. Gas analyzer shows the change in composition, i.e., excess oxygen and no CO in the off gas:



Gas	Concentration
CO	0 ppm
CO_2	5.88 Vol%
O_2	7.23 Vol%

Start feeding green coke at 22 lbs/hr, adjust pressure in CC, furnace temperature drops to 850°C. The flame became yellowish and much brighter when the volatile hits the combustion chamber.

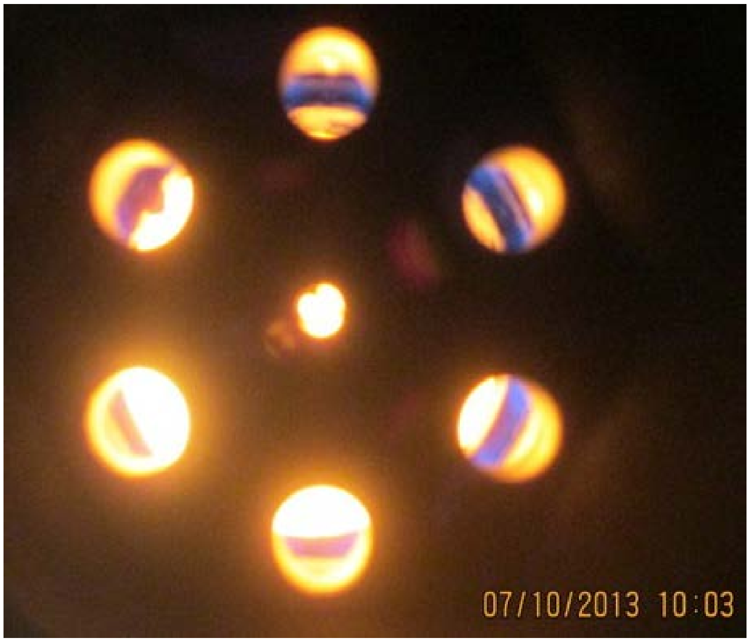

07/10/2013 10:03

Fig 5. Flame in combustion chamber with 22 lbs/hr green coke feeding

After about 10 mins, flame control gives no flame signal and shut off the natural gas (NG), but actually there is still a flame in the combustion chamber. The flame lasted for about 10 mins and finally went off as the combustion chamber cools down without the support of the pilot burner.


07/10/2013 10:07

Fig 6. Flame on the volatile combustion by itself

The flame sensor is a UV sensor, and it is based on the fluctuation of the pilot flame wavelength. When there is a strong combustion, the brighter and more stable combustion flame will cover the pilot flame, so the flame sensor can't see the pilot flame and will send a signal to shut off the NG. Even with good combustion, the pilot burner was shut off and causing this problem. GTI engineers suggested switching to an IR sensor in the future.

- One try to solve the flame sensor problem is to connect the flame control ports 6 and 8, so when the "no flame" warning light is on, it can still power the solenoid valve so NG can still be on. This try is not successful since the flame control actually shut off NG first then turn on the warning light. After restart, the pilot burner went off again, and the gas detector sounds alarm indicating high CO level in the R&D area.
- Another try is to adjust the gas train setting to get better pilot flame. The ratio regulator was adjusted to get better NG to air ratio. This try also failed because of the way how the flame sensor works as mentioned previously.
- Last try is increasing the green coke feeding rate to get more volatile in the combustion chamber. With the assumption that the flame can support itself even without the support of the pilot flame, since there are more volatile available. Throughput was increased from 22 lbs/hr to 50 lbs/hr. This try is also failed, the combustion can't support itself.
- To avoid the effect of condition fluctuation on the gas train, the ratio regulator was removed, and the pressure of NG and air was manually adjusted to get stable pilot flame.

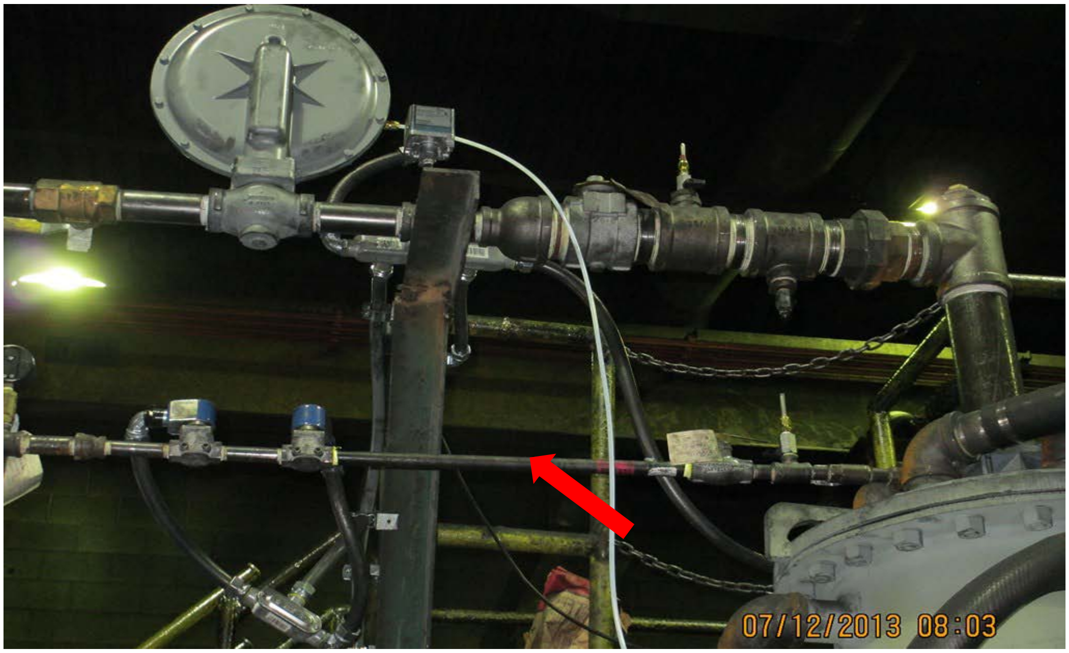


Fig 7. Ratio regulator removed, replaced by a straight pipe

- On July 11, before start the trial again, clean the bottom of the afterburner to see if there are solids in the bottom cone.

Fig 8. Carbon solids in the bottom cone of the afterburner

- Started pilot burner, once furnace temperature reaches 1350 °C, start feeding green coke, at 22 lbs/hr, furnace temperature drops to 1050 °C. No success in increasing throughput, and it is necessary to change the flame control circuit to keep the pilot flame always on in order to maintain stable combustion.

- Changing the flame control by connecting port 3 and 8 and add outside power, so when start the pilot burner, use the outside power to keep the solenoid valve always on. With the pilot flame stay on, the combustion was maintained and we tried all different throughput from 22 lbs/hr to 100 lbs/hr. The furnace is running stable and volatile combustion is complete from the gas analyzer analysis. Furnace is even ready to run 150 lbs/hr, but GTI engineers think data is enough so we stopped the trial. Below is the summary table of the off gas analysis and compressed air settings to combustion chamber 1 and 2.

-

Throughput (lbs/hr)	CO (ppm)	CO_2 (vol%)	O_2 (vol%)	CC1_Compressed Air (SCFM)	CC2_Compressed Air (SCFM)
22	0	7.35	4.35	3	1
35	3	7.77	3.37	3	1
55	14	7.41	4.05	5	3
75	20	7.23	4.19	8	5
100	25	6.61	5.54	11	8

Fig 9. Combustion flame changes with increasing throughput (0→22→35→55→75→100 lbs/hr)

- Furnace flue gas composition changes when the throughput increased, but H_2 is still the major component in the volatile when the calcination temperature is low. This corresponds very well with GTI's lab test data.

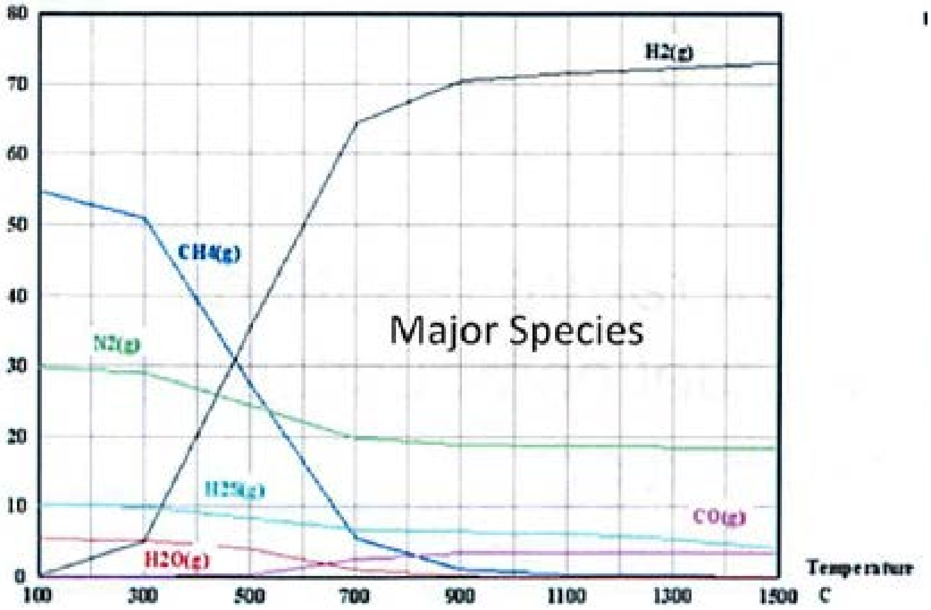


Fig10. GTI flue gas composition lab test result

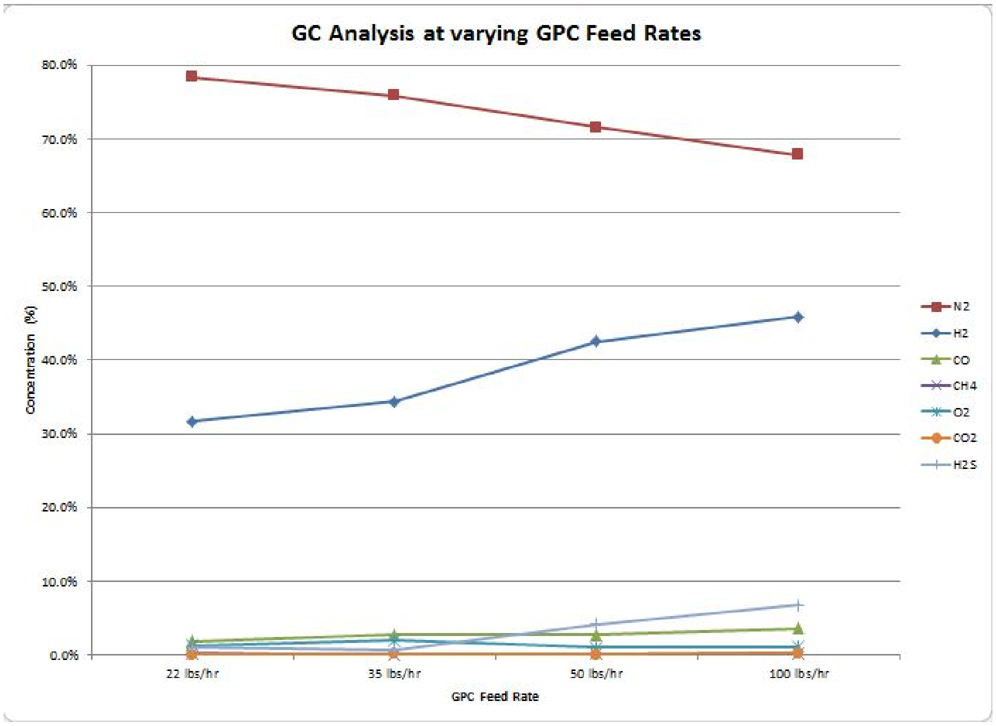


Fig 11. Furnace flue gas composition from GC test. Note: N_2 is included and the percentage is not normalized.

- Smoke & small flame comes out of the tee section cap also periodically according to the green coke feeding.

Fig 12. Smoke comes out of the cap of the tee section

- After trial evaluation. Carbon solids were found in the tee section before entering the horizontal swirling hole, and between afterburner combustion chamber 1 and 2, 2 and 3.

Fig 13. Carbon solids settled inside afterburner, in tee section (left) and between combustion chambers (right).

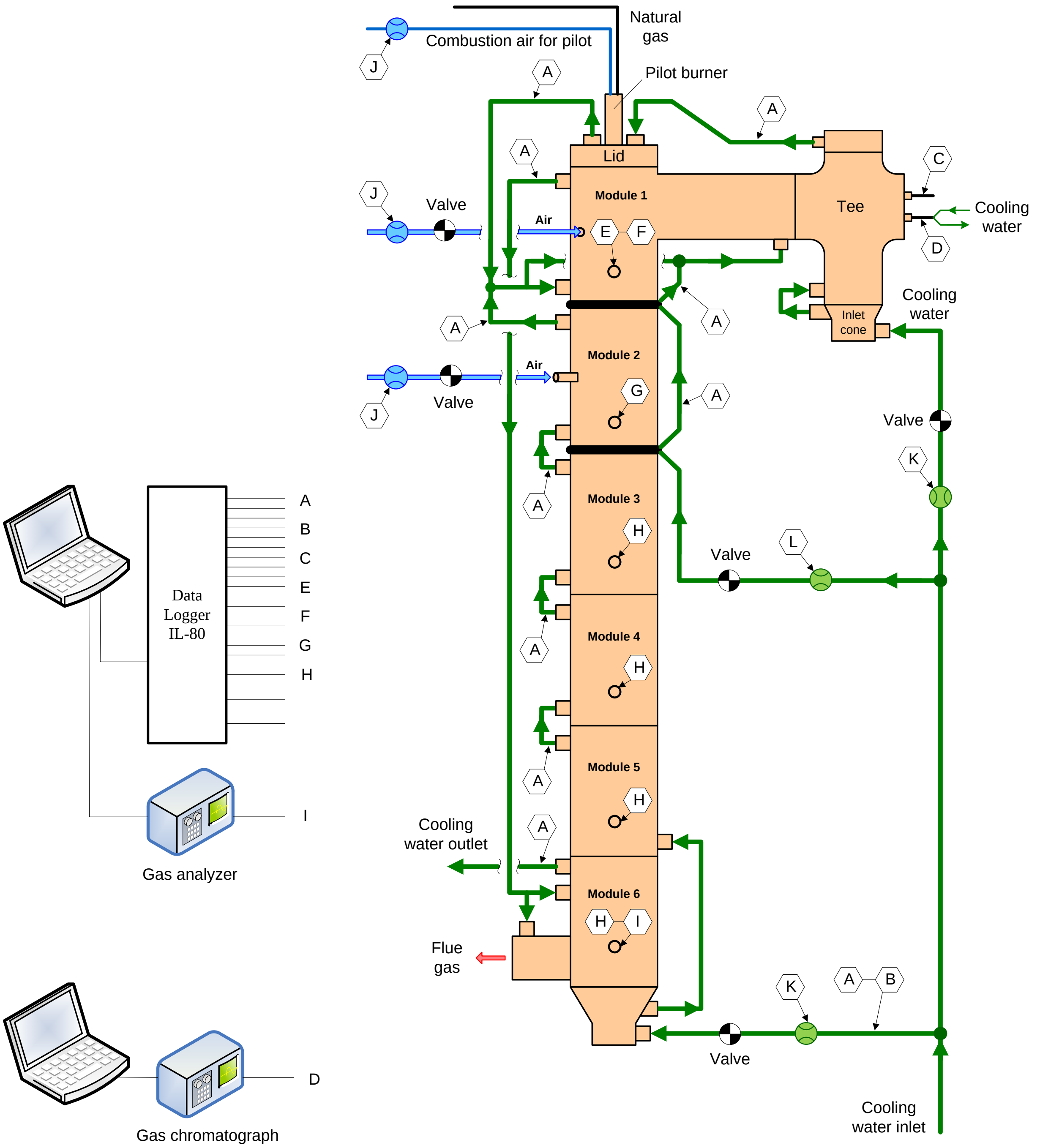
- Solid fines in the system (collected, actually value may be 1.5~2 times collected):
  Settled in tee section=624 g; settled between CC1 and CC2=171 g; CC2 and chamber 3 should have similar settled solid; settle at bottom of afterburner=183.7g.

Major Conclusions:

- With the modification of the system, the trial is a big success. First time successfully run the GTI afterburner for the green coke calcination. Proved the concept of volatile combustion for energy recovery and discovered optimized operation condition for furnace throughput up to 100 lbs/hr.
- It is critical to maintain the pilot burner always on during the trial. According to the discussion with GTI, the system is designed for larger throughput. For less volatile, pilot burner needs to stay on.
- An IR flame sensor was needed in the future for better control of the pilot burner.
- Safety procedure was modified for preventing potential hazards.
- Carbon material accumulated inside the afterburner.
- A collaborated abstract of this work is submitted for consideration of TMS 2014 annual meeting.

APPENDIX G

Measurement system and associated equipment



Pos.	Service	Instrument type	Range	Signal type	Device, model	Quantity	Comments
A	Water temperature	Thermocouple	max 2502°F	K-type TC	Omega KQSS-14U-6 thermocouple	11	¼” O.D., Nominal length 6”
B	Water inlet pressure	Pressure transmitter	0-50 psig	4-20 mA	Setra, model 230	1	Differential, use one side for gauge pressure; 1/4” tubing connection
C	Off-gas temperature	Thermocouple	max 2912°F	S-type TC	Rosemount 1075 Series, Design AK thermocouple	1	22 mm OD, Nominal length 450 mm
D	Off-gas composition	Gas chromatograph			Micro-GC Varian 4000	1	Sample port, 3/8” tubing, water cooled probe
E	Gas temperature in module 1	Thermocouple	max 3272°F	B-type TC	Rosemount 1075 Series, Design AK thermocouple	1	22 mm OD, Nominal length 450mm
F	Gas pressure	Pressure transmitter	+/- 0.5 PSID	4-20 mA	Setra, model 230	1	Differential, use one side for gauge pressure; 1/4” tubing connection
G	Gas temperature in module 2	Thermocouple	max 2912°F	S-type TC	Rosemount 1075 Series, Design AK thermocouple	1	22 mm OD, Nominal length 450mm
H	Gas temperature	Thermocouple		N-type	Omega, cast iron head	4	3/8” OD, 18” length
I	Gas composition	Gas analyzer			Horiba analyzer P250	1	Sample port needed, 3/8” tubing
J	Combustion air flow rate	Flow meter or rotameter/U-tube	0-100 scfm	Specify	Specify	2	Orifice plate if rotameter/U-tube is used
K	Water flow rate	Flow meter or rotameter/U-tube	0-300 GPM	Specify	Specify	2	Orifice plate if rotameter/U-tube is used
L	Water flow rate	Flow meter or rotameter/U-tube	Specify	Specify	Specify	1	Orifice plate if rotameter/U-tube is used