# NOVEL GAS CLEANING/ CONDITIONING FOR INTEGRATED GASIFICATION COMBINED CYCLE

# VOLUME I – CONCEPTUAL COMMERCIAL EVALUATION

### **OPTIONAL PROGRAM FINAL REPORT**

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By

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#### ABSTRACT

Development efforts have been underway for decades to replace dry-gas cleaning technology with humid-gas cleaning technology that would maintain the water vapor content in the raw gas by conducting cleaning at sufficiently high temperature to avoid water vapor condensation and would thus significantly simplify the plant and improve its thermal efficiency. Siemens Power Generation, Inc. conducted a program with the Gas Technology Institute (GTI) to develop a Novel Gas Cleaning process that uses a new type of gas-sorbent contactor, the "filter-reactor". The Filter-Reactor Novel Gas Cleaning process described and evaluated here is in its early stages of development and this evaluation is classified as conceptual. The commercial evaluations have been coupled with integrated Process Development Unit testing performed at a GTI coal gasifier test facility to demonstrate, at sub-scale the process performance capabilities. The commercial evaluations and Process Development Unit test results are presented in Volumes 1 and 2 of this report, respectively.

Two gas cleaning applications with significantly differing gas cleaning requirements were considered in the evaluation: IGCC power generation, and Methanol Synthesis with electric power co-production. For the IGCC power generation application, two sets of gas cleaning requirements were applied, one representing the most stringent "current" gas cleaning requirements, and a second set representing possible, very stringent "future" gas cleaning requirements. Current gas cleaning requirements were used for Methanol Synthesis in the evaluation because these cleaning requirements represent the most stringent of cleaning requirements and the most challenging for the Filter-Reactor Novel Gas Cleaning process.

The scope of the evaluation for each application was:

- Select the configuration for the Filter-Reactor Novel Gas Cleaning Process, the arrangement of the individual gas cleaning stages, and the probable operating conditions of the gas cleaning stages to conceptually satisfy the gas cleaning requirements;
- Estimate process material & energy balances for the major plant sections and for each gas cleaning stage;
- Conceptually size and specify the major gas cleaning process equipment;
- Determine the resulting overall performance of the application;
- Estimate the investment cost and operating cost for each application.

Analogous evaluation steps were applied for each application using conventional gas cleaning technology, and comparison was made to extract the potential benefits, issues, and development needs of the Filter-Reactor Novel Gas Cleaning technology. The gas cleaning process and related gas conditioning steps were also required to meet specifications that address plant environmental emissions, the protection of the gas turbine and other Power Island components, and the protection of the methanol synthesis reactor. Detailed material & energy balances for the gas cleaning applications, coupled with preliminary thermodynamic modeling and laboratory testing of candidate sorbents, identified the probable sorbent types that should be used, their needed operating conditions in each stage, and their required levels of performance.

The study showed that Filter-Reactor Novel Gas Cleaning technology can be configured to address and conceptually meet all of the gas cleaning requirements for IGCC, and that it can potentially overcome several of the conventional IGCC power plant availability issues, resulting in improved power plant thermal efficiency and cost. For IGCC application, Filter-Reactor Novel Gas Cleaning yields 6% greater generating capacity and 2.3 percentage-points greater

efficiency under the Current Standards case, and more than 9% generating capacity increase and 3.6 percentage-points higher efficiency in the Future Standards case. While the conceptual equipment costs are estimated to be only slightly lower for the Filter-Reactor Novel Gas Cleaning processes than for the conventional processes, the improved power plant capacity results in the potential for significant reductions in the plant cost-of-electricity, about 4.5% for the Current Standards case, and more than 7% for the Future Standards case.

For Methanol Synthesis, the Novel Gas Cleaning process scheme again shows the potential for significant advantages over the conventional gas cleaning schemes. The plant generating capacity is increased more than 7% and there is a 2.3 %-point gain in plant thermal efficiency. The Total Capital Requirement is reduced by about 13% and the cost-of-electricity is reduced by almost 9%. For both IGCC Methanol Synthesis cases, there are opportunities to combine some of the filter-reactor polishing stages to simplify the process further to reduce its cost.

This evaluation has devised plausible humid-gas cleaning schemes for the Filter-Reactor Novel Gas Cleaning process that might be applied in IGCC and Methanol Synthesis applications. These schemes are simpler than those used in conventional dry-gas cleaning for these applications and show the conceptual-potential to provide plant availability, plant thermal efficiency and cost improvements over the conventional plants. Since methanol synthesis gas cleaning requirements are more stringent than any other syngas application, the Filter-Reactor Novel Gas Cleaning process is expected to achieve similar advantages for other applications, such as for coal-fueled, Solid Oxide Fuel Cell applications.

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- ACRONYMS AND ABBREVIATIONS AGR Acid Gas Removal
- ASU Air Separation Unit
- COE Cost of Electricity
- EPRI Electric Power Research Institute
- GTI Gas Technology Institute
- HHV Higher Heating Value
- HRSG Heat Recovery Steam Generator
- IGCC Integrated Gasification Combined Cycle
- ISO International Organization for Standards
- KRW Kellogg Rust Westinghouse
- LHV Lower Heating Value
- MDEA Methyldiethanolamine
- PC Pulverized Coal
- PDU Process Development Unit
- PFBC Pressurized Fluidized bed Combustion, -Combustor
- ppbv Parts per billion  $(10^9)$  by volume

- ppmvParts per million by volumeppmwParts per million by weightTCRTotal Capital RequirementVOCVolatile Organic Compound

## 1. EXECUTIVE SUMMARY

The gasification of coal generates a raw gas that requires considerable cleaning, removing particulate and several vapor-phase contaminants to very low levels before the gas can be used in applications such as IGCC power generation or fuel/chemical synthesis. Conventional gas cleaning processes cool the raw gas to a low temperature that results in nearly complete removal of condensable species (primarily water vapor and volatile metal contaminants) from the gas. This produces a condensate stream that is used in a gas-condensate contactor to absorb highly water-soluble contaminants from the gas (halides and ammonia), generating a nearly dry gas and a highly contaminated condensate stream that requires extensive processing. It is followed by "dry-gas" treatment in a low-temperature, gas-solvent absorption contactor to remove sulfur species. In IGCC applications, the clean, dry gas must be rehumidified and diluted with nitrogen to generate a fuel gas that can be fired in turbine combustors with acceptable NOx emission. This "dry-gas" cleaning technology, while being highly effective for gas cleaning, results in a complex process that has high overall power and thermal energy consumption.

Development efforts have been underway for decades to replace this dry-gas cleaning technology with "humid-gas" cleaning technology that would maintain the water vapor content in the raw gas by conducting cleaning at sufficiently high temperature to avoid water vapor condensation and would thus significantly simplify the plant and improve its thermal efficiency. These humid-gas cleaning techniques have been previously designated "hot-gas" or "warm-gas" cleaning. Such methods have the potential for improved thermal efficiency and reduced process complexity, but they have not yet been fully defined for integrated plant operation, nor demonstrated at any significant scale.

The most promising and most developed approach proposed for gas cleaning under humid-gas conditions is to configure a series of stages of gas-sorbent particle contactors that will either adsorb or chemically react with specific contaminants (halide species, sulfur species, mercury species, etc.). For example, sodium-based sorbents have been tested at high temperatures in gas-sorbent contactors to remove halides (primarily HCl), and zinc-, iron-, and a number of other metal oxide-based sorbents have been tested in various types of contactors to remove sulfur species. The types of gas-sorbent contactors considered in the past have been fixed beds, moving beds, fluidized beds, and transport beds, all using appropriate sorbent materials reactive to the specific contaminant of interest and with particle sizes appropriate to the type of contactor. All of these contactors, though, are prone to performance issues related to plugging, transient pressure drop increases, sorbent particle attrition and elutriation, and the need to operate with high-cost, highly durable, specially fabricated sorbent particles. Also, none of these gas-sorbent contactors can achieve the very low gas contaminant levels that will be required in future IGCC plants or the extremely low contaminant levels required in many fuel/chemical synthesis applications, and are only suitable for "bulk" contaminant removal.

#### **Filter-Reactor Novel Gas Cleaning Concept**

Siemens Power Generation, Inc. (Siemens) is conducting a program with the Gas Technology Institute (GTI) to develop a Novel Gas Cleaning process that uses a new type of gas-sorbent contactor, the "filter-reactor". The filter-reactor is both a barrier filter that achieves very efficient removal of particulate from the gas, and a gas-sorbent reactor used for once-through sorbent, gas-contaminant polishing. The filter-reactor behaves, in principle, as a fixed bed reactor, but having several potential advantages over conventional gas-sorbent contactors. It is continuously replenished with fresh sorbent particles by injecting fine sorbent particles into its inlet gas. These sorbent particles distribute uniformly on the filter elements, providing very efficient gas-sorbent contacting conditions, and several contaminant polishing functions, including particulate removal and various gas-phase contaminant removals using once-through sorbents can be simultaneously performed in a single vessel. Two key features of the filter-reactor that contrast it with the conventional gas-sorbent contactors are that its outlet particle loading is extremely low, and it might operate efficiently using cheap, fine, unsupported sorbent particles. The filter-reactor gas-sorbent contactors proposed for use in this highly efficient, humid-gas cleaning process, called the Filter-Reactor Novel Gas Cleaning process, have the potential to provide improved plant operating behavior and improved thermal efficiency while being able to achieve the very low gas contaminant levels that will be required in future IGCC plants or the extremely low contaminant levels required in many fuel/chemical synthesis applications. This process builds upon prior humid-gas cleaning technologies for bulk halide and sulfur removal developed under DOE sponsorship and is integrated with these bulk removal technologies to improve their performance.

#### **Evaluation Objectives and Scope**

The Siemens Novel Gas Cleaning Base Program generated an initial process evaluation supported by laboratory test data that identified the potential merits of advanced technology (Siemens Power Generation, "Novel Gas Cleaning/Conditioning for Integrated Gasification Combined Cycle, Siemens, Base Program Final Report", August, 2001, AC26-99FT40674-02). The overall objective of the Siemens Novel Gas Cleaning Optional Program, described in Volume I of the report, is to produce sub-scale, Process Development Unit (PDU) test data that demonstrates the performance potential of the filterreactor for key humid-gas cleaning and polishing functions. This PDU test data is described and analyzed in Volume II of this Final Optional Program Report. The evaluation described in this report has been conducted to support this program's experimental filter-reactor sub-scale development by:

- devising commercial, integrated, humid-gas cleaning process configurations that apply the filterreactor contacting stages,
- identifying the filter-reactor contacting stages performance requirements,
- identifying their likely ranges of operating conditions,
- generating process material & energy balances and conceptual equipment designs for commercial applications,
- quantifying the overall, conceptually-based, gas cleaning performance and cost potential,
- providing a framework for quantitatively assessing the Process Development Unit (PDU) filterreactor test results generated in this program (reported in Volume II).

The Filter-Reactor Novel Gas Cleaning process described and evaluated here is in its early stages of development and this evaluation is classified as "conceptual." This evaluation is not intended to represent absolute estimates of performance and cost for the plant applications considered, but is only applied to estimate the conceptual performance and cost potential of the Filter-Reactor Novel Gas Cleaning process relative to conventional gas cleaning technologies. A set of process assumptions has been developed for the evaluation that address the features and performance of the Filter-Reactor Novel Gas Cleaning process components based on the current state-of-understanding of those components. Process performance and conceptually-based conclusions for the Filter-Reactor Novel Gas Cleaning potential performance, cost benefits, and technology issues.

Two gas cleaning applications have been considered in the evaluation, the two having significantly differing gas cleaning requirements:

- IGCC power generation,
- Methanol Synthesis with electric power co-production.

For the IGCC power generation application, two sets of gas cleaning requirements have been selected and applied, one representing the most stringent of "current" gas cleaning requirements for IGCC, and a second set representing possible, very stringent "future" gas cleaning requirements for IGCC. The gas cleaning requirements for Methanol Synthesis have been considered in the evaluation because these cleaning requirements represent the most stringent of cleaning requirements and the most challenging for the Filter-Reactor Novel Gas Cleaning process.

The scope of the evaluation for each application was:

- Select the configuration for the Filter-Reactor Novel Gas Cleaning Process, the arrangement of the individual gas cleaning stages, and the probable operating conditions of the gas cleaning stages to conceptually satisfy the gas cleaning requirements;
- Estimate process material & energy balances for the major plant sections and for each gas cleaning stage;
- Conceptually size and specify the major gas cleaning process equipment;
- Determine the resulting overall performance of the application:
  - generating capacity and heat rate,
  - product and by-product production rates,
  - chemical and catalyst feed rates and auxiliary consumptions,
  - plant emissions performance,
  - gas turbine protection performance,
  - synthesis catalyst protection performance,
  - quantities of, and final fate of gas contaminants and waste solids.
  - Estimate the investment cost and operating cost for each application:
    - gas cleaning process major equipment purchased and installed cost,
    - plant Total Capital Requirement,
    - plant annual operating cost,
    - plant Cost-of-Electricity.

Analogous evaluation steps were applied for each application using conventional gas cleaning technology, and comparison was made to extract the potential benefits, issues, and development needs of the Filter-Reactor Novel Gas Cleaning technology.

## **Evaluation Basis**

Two types of applications for Filter-Reactor Novel Gas Cleaning have been considered: 1) IGCC power generation with two levels of gas cleaning requirements, Current Standards and Future Standards; and 2) Methanol Synthesis with co-production of power, subjecting the Filter-Reactor Novel Gas Cleaning process to very stringent requirements. A set of commercial plant specifications are presented in Section 3 of this report that identify the evaluation bases for these two applications in sufficient detail to conduct the conceptual evaluation.

The major factors selected for the plant basis as being representative of likely future plants are:

- gasifier type: representative of a single-stage, oxygen-blown, entrained, slagging gasifier,
- coal feeding and pressurization method: coal-water slurry system,
- air-side integration level: 100% integration (even though 50% may be nearer the optimum condition),
- coal type: Illinois Number 6, high-sulfur bituminous,
- raw-gas heat recovery method: radiant and convective heat recovery to generate HP-steam,
- gas turbine type: representative of "F" turbine technology (SGT6-5000F), modified for air-side integration and syngas operating duty,
- methanol production rate: 218 tonne/day (240 tons/day),
- methanol grade: fuel-grade.

The gas cleaning process and related gas conditioning steps must meet specifications that address:

- the plant environmental emissions,
- the protection of the gas turbine and other Power Island components,
- the protection of the methanol synthesis reactor.

The following gas cleaning specifications are of critical importance to the design of the gas cleaning process, and achieving these specifications, while maintaining the overall plant performance and economics at acceptable levels, is a key measure of the success of the gas cleaning technology.

The IGCC power plant evaluation considered two sets of gas cleaning standards, "Current Standards" and "Future Standards" with respect to environmental emissions and Power Island protection standards. Current Standards are based on the "best-of-current-practice" reported for existing IGCC power plants. The IGCC power plant Future Standards approach the emissions performance of natural gas-fired power plants, and reflect standards that may be imposed on the future generation of IGCC plants. The gas cleaning requirements considered for the Methanol Synthesis plant evaluation include standards for the protection of the methanol synthesis reactor as well as emissions and Power Island standards. The environmental standards applied for Methanol Synthesis are identical to the Future Standards case used for IGCC. The Power Island standards are the same as those used for the IGCC application. The Methanol Synthesis standards for gas cleaning are extremely stringent. These gas cleaning standards are listed and compared in Table 1.1.

Evaluation Case	IGCC		Methanol
Standards	Current	Future	Future
Environmental			
SOx, % coal sulfur removed	99	99.98	99.98
NOx, ppmv at stack, corrected to 15% oxygen, dry	15	5	5
Particulate, mg/MJ (lb/10 <sup>6</sup> Btu)	2.15 (0.005)	2.15 (0.005)	2.15 (0.005)
Mercury, % coal Hg removed	90	95	95
Power Island fuel gas			
Halides, ppmv	5	5	5
volatile metals, ppbv	40	40	40
Particulate, ppmw	0.1	0.1	0.1
Methanol Synthesis syngas			
total sulfur species, ppbv			60
total halide species, ppbv			10
Ammonia, ppmv			10
hydrogen cyanide, ppbv			10
Metal carbonyls, ppbv			100
Particulate, ppmw			0.1
Mercury, % removal			95

 Table 1.1 – Gas Cleaning Requirements Basis for Evaluation Cases

#### **IGCC Evaluation Results**

The Filter-Reactor Novel Gas Cleaning technology can be configured to address and conceptually meet all of the gas cleaning requirements for IGCC. It can also potentially overcome several of the conventional IGCC power plant availability issues, and can result in improved power plant thermal efficiency and cost.

Overall, generic gas cleaning process schemes for IGCC using conventional dry-gas cleaning and Filter-Reactor Novel Gas Cleaning are illustrated and compared in Figure 1.1. The AGR Absorber in the figure is a generic process block representing any one of a number of technologies, such as MDEA absorption or Rectisol absorption. The conventional dry-gas cleaning process for IGCC contains a series of highly-integrated process steps with several recycle streams and applies numerous heat interchangers that provide temperature control and water vapor condensation and gas re-humidification to generate a clean fuel gas. Raw gas cooling is followed by particle removal and COS hydrolysis in both process schemes. Conventional dry-gas cleaning applied low-temperature cooling to condense out halide and

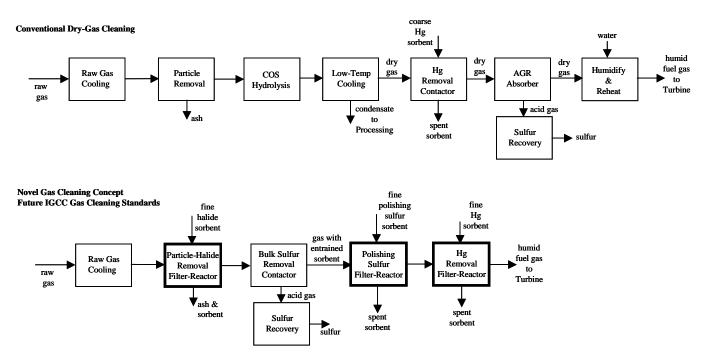


Figure 1.1 – Conventional and Novel Gas Cleaning Process Schemes for IGCC

ammonia. Mercury removal is performed in a low-temperature, packed bed adsorber process. An absorber process is used for acid gas removal (AGR) and its selection depends on the degree of sulfur removal required. The cleaned syngas is humidified and reheated before passing to the gas turbine.

The Filter-Reactor Novel Gas Cleaning scheme is relatively simple compared to the conventional dry-gas cleaning process. In the Filter-Reactor Novel Gas Cleaning process, halides are controlled by once-through nahcolite, or trona sorbent injection into a filter-reactor located before the bulk sulfur removal process. Bulk sulfur removal is conducted in a transport desulfurizer using zinc-based sorbent. A filter-reactor following the transport reactor captures entrained sulfur sorbent from the transport reactor and provides conditions for additional reaction between the entrained sorbent and gas, with the temperature of the filter-reactor controlled to provide best reaction conditions. Additional sorbent is injected into this filter-reactor to achieve the IGCC Future Standards for sulfur removal. Mercury is captured in a final filter-reactor using a humid-gas mercury sorbent, although these two cleaning functions could potentially be performed in the same filter-reactor vessel if an appropriate mercury sorbent could be identified. NOx emission control is by means of advanced, low-NOx staged, turbine-combustors that incorporate ammonia decomposition.

While the Filter-Reactor Novel Gas Cleaning process should be able to meet all of the gas cleaning requirements, the conventional dry-gas cleaning technology, using MDEA Acid Gas Removal for Current Standards, and Rectisol Acid Gas Removal for Future Standards, will result in lower halide content and lower fuel-bound nitrogen (ammonia and HCN) in the clean fuel gas.

The IGCC overall conceptual performance results are tabulated in Table 1.2. The results indicate that the Filter-Reactor Novel Gas Cleaning scheme has the potential for significant improvements in IGCC power plant generating capacity and heat rate. Filter-Reactor Novel Gas Cleaning yields 6% greater generating capacity and 2.3 percentage-points greater efficiency under the Current Standards case, and more than 9% generating capacity increase and 3.6 percentage-points higher efficiency in the Future Standards case. Note that Novel Gas Cleaning performance is almost entirely insensitive to the gas cleaning standards, with little difference between the Current and Future Standards cases. Solid waste from IGCC with Filter-Reactor Novel Gas Cleaning is about 8% greater than IGCC with conventional

dry-gas cleaning. While the conceptual equipment costs are estimated to be only slightly lower for the Filter-Reactor Novel Gas Cleaning processes than for the conventional processes, the improved power plant capacity results in the potential for significant reductions in the plant cost-of-electricity, about 4.5% for the Current Standards case, and more than 7 % for the Future Standards case.

Gas Cleaning Technology	Conventional Cleaning	Novel Cleaning	Conventional Cleaning	Novel Cleaning
Gas Cleaning Standards	Current	Current	Future	Future
Generation capacity, MWe	285	303	276	303
Plant thermal efficiency (%, HHV)	37.6	39.9	36.3	39.9
Plant Heat Rate (HHV),	9,574	9,022	9,916	9,022
kJ/kWh (Btu/kWh)	(9,074)	(8,551)	(9,399)	(8,551)
Total Capital Requirement, \$/kW	1,500	1,415	1,614	1,435
Total COE, cents/kWh	6.6	6.3	6.9	6.4

 Table 1.2 – Conventional and Novel Gas Cleaning Performance and Cost for IGCC

## **Methanol Synthesis Evaluation Results**

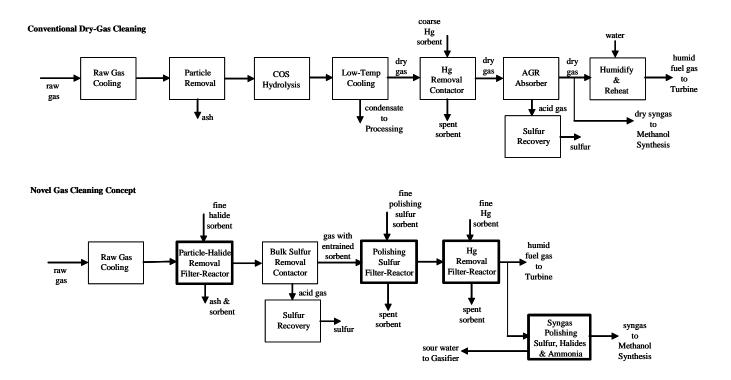
The chemical synthesis application evaluated is a combined electric power and Methanol Synthesis plant, so there are two major gas streams be cleaned: a fuel gas stream for power generation, and a syngas stream for Methanol Synthesis. The Filter-Reactor Novel Gas Cleaning technology can be configured to meet all of the gas cleaning requirements for gas cleaning presented by Methanol Synthesis.

The primary gas cleaning process features applied for conventional dry-gas cleaning and for Filter-Reactor Novel Gas Cleaning are:

- conventional dry-gas cleaning for Methanol Synthesis is similar to the IGCC gas cleaning process for Future Standards, but uses Rectisol Acid Gas Removal technology, as well as fixed, guard-bed sulfur sorbent stages to meet the very stringent gas cleaning requirement for sulfur control;
- Filter-Reactor Novel Gas Cleaning is similar to the IGCC gas cleaning process for Future Standards, but incorporates an additional filter-reactor sulfur polishing stage and applies a water scrubbing step for methanol synthesis syngas halide and ammonia polishing.

The two process schemes are illustrated and compared in Figure 1.2. The conventional dry-gas cleaning process uses low-temperature wet scrubbing for halide and ammonia removal, Rectisol Acid Gas Removal process for sulfur species control, and low-temperature packed bed adsorption of mercury. It then separates the gas into a methanol syngas stream and a fuel gas stream, and it places guard beds within the Methanol Synthesis process for final-stage syngas sulfur removal and metal carbonyls removal.

The Filter-Reactor Novel Gas Cleaning process uses halide sorbent injection into a Filter-Reactor for particulate and bulk halide removal, a zinc oxide-based sorbent for bulk sulfur removal, and a Filter-Reactor sulfur polishing stage to meet the IGCC fuel gas cleaning requirements. A stage of mercury adsorbent injection into a mercury Filter-Reactor is operated at a temperature of 260°C (500°F) to achieve the mercury requirement. The separated methanol syngas stream is then treated in an additional sulfur polishing Filter-Reactor, followed by a water scrubbing stage for syngas polishing of halides and ammonia. The sour water from the scrubber is recycled to the gasifier where the captured ammonia is thermally decomposed, and the captured halide is released and eventually captured in the bulk halide removal Filter-Reactor, making this water scrubber process much less complex and less costly than that used in the conventional dry-gas cleaning process.



#### Figure 1.2 – Conventional and Novel Gas Cleaning Process Schemes for Methanol Synthesis

The Novel Gas Cleaning process scheme again shows the potential for significant advantages over the conventional gas cleaning schemes (Table 1.3). The plant generating capacity is increased more than 7% and there is a 2.3 %-point gain in plant thermal efficiency. The Total Capital Requirement is reduced by about 13% and the cost-of-electricity is reduced by almost 9%. Again, there are opportunities to combine some of the filter-reactor polishing stages to simplify the process further to reduce its cost.

Gas Cleaning Technology	Conventional	Novel
	Cleaning	Cleaning
Generation capacity, MWe	288	309
Plant thermal efficiency (%, HHV)	32.7	35.0
Plant Heat Rate (HHV),	11,008	10,286
kJ/kWh (Btu/kWh)	(10,434)	(9,749)
Total Capital Requirement, \$/kW	1,791	1,565
Total COE, cents/kWh (constant \$)	5.6	5.1

Table 1.3 – Conventional and Novel Gas Cleaning Performance and Cost for Methanol Synthesis

#### **Evaluation Conclusions**

This evaluation has devised plausible humid-gas cleaning schemes for the Filter-Reactor Novel Gas Cleaning process that might be applied in IGCC and Methanol Synthesis applications. These schemes are simpler than those used in conventional dry-gas cleaning for these applications and show the conceptual-potential to provide plant availability, plant thermal efficiency and cost improvements over the conventional plants. Since methanol synthesis gas cleaning requirements are more stringent than any other syngas application, the Filter-Reactor Novel Gas Cleaning process is expected to achieve similar advantages for other applications, such as for coal-fueled, Solid Oxide Fuel Cell applications.

The Filter-Reactor should have a basic design similar to near-commercial barrier filters, with a large number of independently pulse-cleaned filter plenums that will allow the Filter-Reactor to maintain high levels of emission control. Sorbent particle sizes injected into the Filter-Reactors are expected to

operate best at -325 mesh, with a mass-mean size of about 20  $\mu$ m. The major uncertainties are 1) the contaminant removal performance that can actually be achieved in these Filter-Reactors, with their relatively thin, 5-13 mm (0.2 to 0.5 inch) sorbent filter cakes and low gas velocities through the filter cakes, and 2) the possible reaction-sintering behavior of the filter cakes that might occur at the stage conditions. These uncertainties were resolved in PDU testing conducted under representative conditions in this program.

Detailed material & energy balances for the gas cleaning applications, coupled with preliminary thermodynamic modeling and laboratory testing of candidate sorbents have identified the probable sorbent types that should be used, their needed operating conditions in each stage, and their required levels of performance. These performance goals and the results from the PDU testing are summarized in Table 1.4. In general, the performance goals have been demonstrated in the PDU testing, with the exceptions noted in the table. A water scrubbing stage is used for syngas polishing of halides and ammonia in the Methanol Synthesis application, and this stage should be able to be applied commercial using available technology experience. The conditions and performance levels that have not been demonstrated in the PDU testing in this program have been extrapolated from the PDU test results to apply to the commercial design and evaluations. Particle removal efficiency was not measured in this test program because of the previously established success of barrier filter technology

The evaluation utilized a regenerative, zinc-based sulfur sorbent in a transport reactor configuration for bulk sulfur removal, but the Filter-Reactor Novel Gas Cleaning process can be coupled with any developing bulk desulfurization technology (alternative sorbents and alternative gas-sorbent bulk desulfurization contactors) operating under humid gas cleaning conditions. The use of alternative bulk desulfurization technology will alter some of the Filter-Reactor stage conditions and polishing sorbent requirements.

Cleaning Stage	Sorbent type	Process Temperature	Process Performance Goals	PDU Test Results (Final Report Volume II)
Stuge	(-325 mesh)	°C (°F)	Gouis	(I mui Report Volume II)
IGCC Appl	ications			
Bulk halide removal	Sodium mineral (Trona or Nahcolite)	593 (1100)	<ul><li>99% halide removal,</li><li>5 ppmv HCl outlet,</li><li>Na/Cl mole feed ratio 4,</li><li>75% ammonia decomposition.</li></ul>	Met the performance goals at 427°C (800°F), Ammonia decomposition not measured (not in program scope)
Sulfur polishing	Zinc-titanate	482 (900)	96% removal, 40 ppmv inlet to 2 ppmv outlet, Zn/S mole feed ratio 3.	Not considered in PDU tests (focus placed on more challenging Methanol sulfur polishing)
Mercury removal	TDA sorbent	204-316 (400-600)	90-95% Hg removal, Sorbent/Hg mass feed ratio 1000, Possibly simultaneous with sulfur polishing.	90% Hg removal achieved at 260°C (500°F), Simultaneous sulfur removal not attempted (insufficient test time).
Methanol S	ynthesis Applic	ation		
Halide & ammonia polishing	Water absorbent	93-149 inlet (200-300)	97% ammonia removal to 10 ppmv, 99.8% HCl removal to 10 ppbv.	Halide and ammonia scrubbing not addressed in PDU tests (design from scrubbing experience).
Sulfur polishing	Zinc-titanate	260-316 (500-600)	<ul><li>98% sulfur removal,</li><li>60 ppbv sulfur outlet,</li><li>Zn/S mole ratio 5.</li></ul>	Sulfur polishing met the performance goals in PDU tests.
Mercury removal	TDA sorbent	204-316 (400-600)	95% Hg removal, Sorbent/Hg mass feed ratio 1000.	90% Hg removal achieved at 260°C (500°F) (data extrapolated for commercial evaluation).

 Table 1.4 – Filter-Reactor Novel Gas Cleaning Stage Performance Goals and Test Results

## 2. INTRODUCTION

IGCC is a relatively recently-commercialized power generation technology. It competes with conventional pulverized coal (PC) steam plant power generation, as well as with natural gas combinedcycle power generation. IGCC power generation applies conventional, low-temperature gas cleaning technology and has demonstrated its environmental emission control capabilities, being far superior to other coal-fueled power generation technologies. IGCC is designated by many as the coal-based power generation technology of the future because of its excellent environmental performance, its ability to utilize a range of solid and liquid fossil fuels, and its ability to be easily adapted for  $CO_2$  sequestration with relatively low cost impact.

IGCC power generation marketability, though, is currently hampered by its process complexity, high capital and maintenance costs, and low power plant availability, in large-part due to the characteristics of conventional, low-temperature gas cleaning technology. Many aspects of IGCC with conventional, low-temperature fuel gas cleaning technologies are still being refined and upgraded to provide more acceptable power plant availability and cost. Five factors inherent in conventional, low-temperature, dry-gas cleaning technologies reduce IGCC power plant efficiencies and increase equipment costs:

- 1) Nearly all of the water vapor in the raw gas is condensed and removed, resulting in a significant plant energy loss;
- 2) The low-temperature gas cooling calls for the use of several stages of complex and expensive process stream heat interchangers, and with some acid gas removal technologies, solvent chilling or even refrigeration is needed that consumes significant energy;
- 3) The process condensate streams generated by raw gas condensation require processing to effectively remove their contained contaminant salts and dissolved gases and prepare them for final disposition, increasing plant complexity and cost;
- 4) The cleaned and reheated fuel gas has a high peak flame temperature with large NOx generationpotential in the gas turbine combustors, and it must be diluted by water vapor, using a fuel gas humidification process or steam injection, resulting in additional plant complexity;
- 5) The low-temperature sulfur absorption processes used remove a significant portion of the fuel gas CO<sub>2</sub> content along with the sulfur species, reducing the fuel gas mass flow and making sulfur recovery more energy intensive and expensive.

Conventional, low-temperature dry-gas cleaning technology can generally meet all of the host of gas cleaning requirements that are imposed for gas turbine protection and plant emissions control. Several of the IGCC plant availability issues, though, that have been reported in the literature relate directly to the nature of the conventional, low-temperature, dry-gas cleaning technology:

- Volatile metal species in the cleaned fuel gas, in the form of iron and nickel carbonyls, are reported in some IGCC power plants, resulting in deposition and corrosion in the gas turbine combustors, and disruption of plant operation (Collodi and Brkic, 2003; Bonzani and Pollarolo, 2004; Bruijn et al, 2003). The conventional gas cleaning process operating conditions may promote the formation of these metal carbonyls and protection from these metal carbonyls is not generally included in IGCC these carbonyls are commonly guarded against in Methanol Synthesis plants by installing low-temperature, packed bed carbonyl adsorbers before the methanol synthesis reactor.
- Particulate, generated by fuel gas piping corrosion and by heat exchanger leaks, or entering the gas through ineffective particulate control devices, is reported in some IGCC power plants to reduce availability, resulting in gas cleaning process equipment fouling, and gas turbine erosion and deposition. This has been dealt with in some IGCC power plants by adding intermediate fuel gas particulate control to protect equipment from such particulate (McDaniel and Hornick, 2003).
- The process condensate streams generated in the conventional, low-temperature gas cleaning processes are highly corrosive to process equipment and result in reduced power plant availability.

Coal-gasification-based Methanol Synthesis has been operating commercially for many years at the Eastman Chemical Company using conventional, low-temperature dry syngas cleaning to meet the stringent syngas cleaning requirements for Methanol Synthesis (Air Products, 2003; Wang, 1997). Here, the gas cleaning process is designed to meet all of the cleaning requirements of the gas turbine, the plant environmental emissions, and the Methanol Synthesis process. This requires significantly different, more effective syngas desulfurization technologies to be applied than are used in IGCC, and the Rectisol Acid Gas Removal process has been applied because of its ability to achieve very low sulfur content in the synthesis gas (Smith, 2000). To ensure acceptable plant availability, the Methanol Synthesis catalyst is protected by guard beds for additional sulfur and metal carbonyl removal.

## Filter-Reactor Novel Gas Cleaning Concept

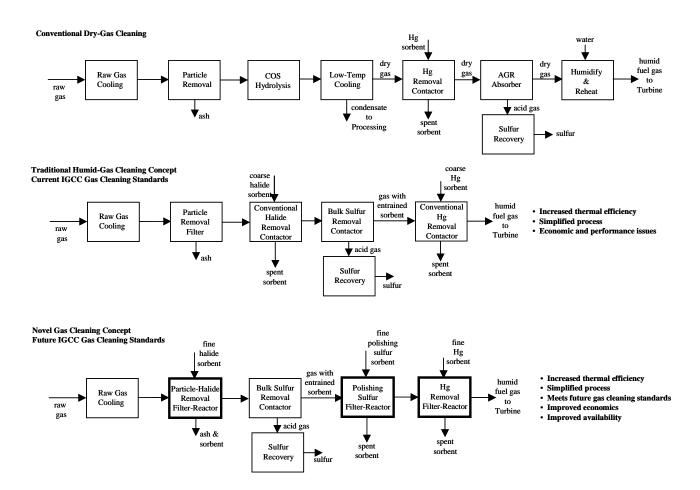
The basic principles of the Filter-Reactor Novel Gas Cleaning technology for IGCC and Methanol Synthesis, as well as for other fuel or chemical synthesis applications are described below. The gas contaminants of major interest to IGCC and Methanol Synthesis are coal ash particles (in slag and char forms), sulfur species, halides, fuel-bound nitrogen (such as ammonia and HCN), volatile metals, and mercury. Each of these contaminants exist at differing levels in the raw gas and need to be reduced by a differing process technique to meet clean gas requirements. For example, sulfur species generally exist in relatively large quantities in the raw gas (typically, total sulfur is as high as 10,000 ppmv) and must be reduced by a large factor of about 99% or greater. At the other extreme, mercury exists in very small quantity (typically, about 3 ppbv mercury in the raw gas) and needs to be reduced by a relatively small factor of 90-95%.

#### Filter-Reactor Novel Gas Cleaning Process Concept

The conceptual configuration of the conventional, dry-gas cleaning process; the traditional, advanced humid-gas cleaning process; and the Filter-Reactor Novel Gas Cleaning, humid-gas cleaning process are illustrated in Figure 2.1. In each of the three process configurations, the raw, high-temperature gas issued from the gasifier is first cooled in a raw gas cooler. The gas is then cleaned of particulate using cyclones and a particle barrier filter. In the conventional, dry-gas cleaning configuration, the gas is then cooled to a low-temperature, condensing out most of the water vapor in the gas and removing most of the halides and ammonia contained in the gas in condensate-gas contacting columns. Mercury is then removed from the dry-gas in a adsorbent-gas contactor, such as a fixed bed reactor. The gas then passes through an appropriate, conventional acid gas removal process (e.g., MDEA), a solvent-gas column absorber and solvent stripper column, to reduce the sulfur species content to a sufficient level. In some cases, the gas COS content must first be hydrolyzed to H<sub>2</sub>S so that sufficiently low sulfur levels can be achieved. The sulfur species removed from the gas is converted to elemental sulfur in a conventional Claus plant. The cold, dry-gas is then reheated and humidified before being fired in the gas turbine.

The traditional, advanced humid-gas cleaning processes operate at temperatures warm enough that no sour condensate is generated (>260°C). Following particle removal, the gas is contacted with halide sorbent in a conventional contactor, such as a fixed bed, moving bed, or a fluidized bed reactor, capturing the halides as solid reaction products for disposal. The humid gas is then desulfurized in a gas-sulfur coarse-sorbent contactor, such as a moving bed, fluidized bed, or transport bed. The sulfur sorbent is regenerative, and regenerated sulfur is converted to elemental sulfur using modified-Claus plant operation. A number of sulfur sorbents have been under development to achieve very efficient sulfur removal at temperatures between 260 and 538°C (500 and 1000°F), with ZnO-based sorbents having the highest potential. Mercury removal in a gas-sorbent contactor is then performed, using an advanced, coarse mercury sorbent that operates at a temperature of at least 245°C (400°F), in a gas-sorbent contactor such as a fixed bed or fluidized bed. The clean, humid gas is then reheated and fired in the gas turbine. The cleanup process, operating under humid-gas conditions, allows the IGCC power plant to operate at higher thermal efficiency, but issues with the durability of the sulfur sorbent, the mercury sorbent, process

availability, and the cleanliness of the fuel gas (particulate content, fuel-nitrogen content) and its ability to achieve the very stringent cleaning standards for Methanol Synthesis exist.



## Figure 2.1 – Comparison of Filter-Reactor Novel Gas Cleaning Process with Traditional Humid-Gas Cleaning and Conventional Dry-Gas Cleaning Processes

The conceptual features of the Filter-Reactor Novel Gas Cleaning Process for IGCC fuel gas and Methanol Synthesis and other Chemical Synthesis syngas cleaning applications are:

- As in the traditional humid-gas cleaning process, gas condensation is avoided, or minimized, reducing the gas temperature only as low as is needed for the dry sorbent particle-contaminant removal reactions to effectively proceed.
- The process uses a series of gas, contaminant-removal stages to achieve the levels of contaminants in the gas required by the application. For contaminants that exist in a relatively large quantity in the raw gas (i.e., sulfur and halides), the initial stage removes the "bulk" of the contaminant. Subsequent polishing in "filter-reactor" steps are used to achieve levels meeting the gas cleaning requirements. Each subsequent stage removes a much smaller amount and operates at a temperature lower than the previous stage. This staged arrangement is used because higher operating temperature induces a higher sorbent-contaminant reaction rate, but the higher-temperature sorbent-contaminant reaction thermodynamics limit the outlet concentration of the contaminant that can be achieved. Lower operating temperature, in contrast, reduces the sorbent-contaminant reaction rate, but the lower-temperature sorbent thermodynamics provides the potential for lower outlet contaminant

concentration. The temperature of each removal stage is selected based on the contaminant removal nature of the sorbent particles to be used in each stage and the contaminant outlet concentration that is desired. This staged arrangement is inherently superior to single-stage, single-temperature processes that attempt to achieve contaminant control of such contaminant species.

- The bulk sulfur removal stage can be one of many bulk desulfurization processes currently under development that use fixed beds, fluidized beds or transport reactors with appropriate sorbent or catalyst particles, having forms such as regenerative zinc-, iron-, copper-, manganese-based sorbents. The Filter-Reactor Novel Gas Cleaning Process integrates well with these bulk sulfur removal processes and enhances their performance by capturing and utilizing sorbent particles that are inevitably lost from these bulk desulfurizers, being carried out with the exiting fuel gas.
- For contaminants existing in very small quantity in the raw gas and requiring relatively small removal factors (i.e., mercury), a single stage of removal is ample, but this stage must operate at low enough temperature so that thermodynamics are favorable to the desired outlet contaminant concentration.
- The process uses small-diameter sorbent particles (nominal feed size is <325 mesh, with mean size about 20 microns, but feed sizes up to 100 microns are potentially effective) to remove the contaminants by chemical and/or physical adsorption reactions.
- Multiple contaminant removal can be performed in a single stage if desired. For example, sulfur and halides can be removed simultaneously by injecting appropriate sorbents into the same filter-reactor vessel with appropriate selection of the operating temperature.
- In principle, the only difference between IGCC fuel gas cleaning, and the more stringently controlled Chemical Synthesis applications will be the number of gas cleaning stages and the conditions of those stages.

## Filter-Reactor Gas-Sorbent Contactor Concept

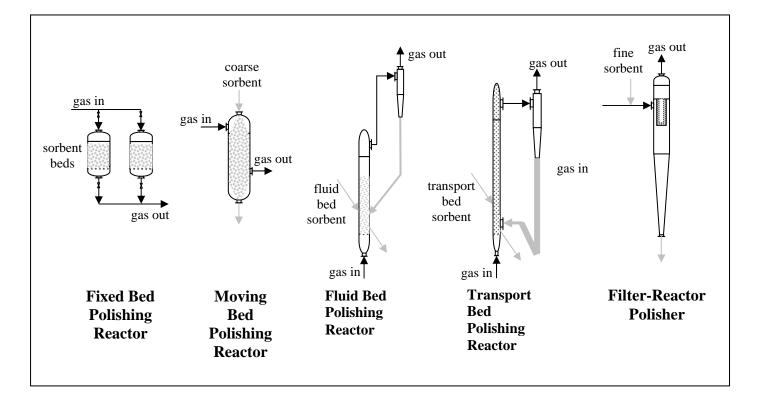
The filter-reactor, gas-sorbent contactor is a substitute for more conventional gas-sorbent contactors (fixed beds, moving beds, fluidized beds, transport beds) used for contaminant polishing, and these are illustrated in Figure 2.2, with the reactors shown at their relative size scales. In these contaminant polishers the gas-phase contaminants are removed by particulate-sorbent reactions with the contaminants, forming stable, solid-phase reaction products. In most cases, the sorbents are used on a once-through basis with no regeneration and the contaminants are relatively small in their content with little reaction heat effects, and these are designated "polishing" rather than "bulk" contaminant removal.

The Filter-Reactor features are:

- Since the clean gas must be essentially particulate-free, barrier filters are used as the sorbent reactors in the gas cleaning process -- all other forms of sorbent-contaminant reactors (fixed beds, moving beds, fluidized beds, transport beds) result in particulate contamination of the exiting gas.
- On each stage of the process, sorbent particles are injected into the inlet piping of a barrier filter vessel. The barrier filter unit effectively separates all of the injected sorbent particles from the gas, as well as removing any other contaminant particles existing in the gas. This "filter-reactor" is the only type of reactor that can provide near-zero outlet particulate content while operating in a near-steady, continuous fashion. The filter-reactor provides an ideal reaction environment for the injected sorbent particles. The sorbent particles mix vigorously with the gas and reside as a dilute mixture of sorbent particles and gas for 1 to 10 seconds before the sorbent particles deposit on the filter elements in the vessel to form a filter cake. This filter cake acts as a fixed bed reactor as the gas passes through it, and the contact time between the sorbent filter cake and the gas is 0.1 to 1 second. The filter cake continually grows in thickness as more sorbent is deposited, and periodically (ever 15 minutes to every 100 hours depending on the contaminant content in the gas) the cake is removed by a reverse gas pulse, and the buildup of a new filter cake begins.
- Barrier filters can be utilized as semi-continuous, fixed bed reactors, designated "filter-reactors", having extremely effective particle sorbent-contaminant removal performance and having no need for

parallel reactor vessels with complex switching valves and piping mechanisms that are used with fixed bed reactors.

- Multiple contaminants can be simultaneous removed in a single filter-reactor if appropriate sorbents effective for the contaminant species removal at the same temperature can be identified.
- Powdered sorbent particles suitable for injection into filter-reactors have very high specific surface area on the filter cakes, and have no need for highly reactive support structure, or special particle attrition resistance features, so they are expected to be relatively cheap and highly available.



## Figure 2.2 – Filter-Reactor Comparison with Conventional Contaminant Polishing Reactors

The characteristics of the various types of gas-sorbent contactors for gas polishing are listed in Table 2.1. The filter-reactor has several advantages over the more conventional fixed bed reactor configuration commonly used for gas-contaminant polishing. The conventional fixed bed reactor, if applied for halide, sulfur, and mercury removal applications might work well as a contaminant removal reactor, but is prone to the following practical issues:

• The fixed bed reactor is a batch reactor that requires periodic isolation from the process, depressurization and cooling, sorbent removal, sorbent refill, repressurization, re-integration with the process gas and reheat. This can be done with one, or more, parallel reactor vessels that are periodically switched in operation from adsorption to regeneration and/or recharge using switching valves and bypass piping.

Contactor	Merits	Issues
type Fixed bed	<ul> <li>No solids transport required</li> <li>Unattended, simple operation</li> <li>Little bed bypassing</li> <li>Best traditional contactor for gas polishing under low heat-generation conditions</li> </ul>	<ul> <li>Multiple, parallel vessels needed with switching valves</li> <li>Bed pressure drop increases and plugging – need particle- free inlet gas</li> <li>Need excess capacity design</li> <li>Large pressure drop vs. vessel size trade-off</li> <li>Some particle attrition and elutriation</li> <li>Need bed support, inlet gas distribution, bed loading and unloading features and facilities</li> <li>No on-line control and difficult for multiple contaminant removal</li> <li>Large particles needed with large mass transfer resistance</li> <li>Large inventory of sorbert exposed to gas</li> </ul>
Moving bed	<ul> <li>Yields steady-state operation with some control capability</li> <li>More tolerant of heat generation than the fixed bed</li> </ul>	<ul> <li>Large inventory of sorbent exposed to gas</li> <li>Complex bed flow and handling equipment</li> <li>Non-uniform gas flow</li> <li>Large particles needed with large mass transfer resistance</li> <li>Large inventory of sorbent exposed to gas</li> <li>Bed pressure drop increases and plugging – need particle-free inlet gas</li> <li>Large pressure drop vs. vessel size trade-off</li> <li>Some particle attrition and elutriation</li> </ul>
Fluid bed Transport bed	<ul> <li>Easy sorbent feeding and withdrawal</li> <li>Can control performance by sorbent feed rate</li> <li>Uniform temperatures</li> <li>Small vessel diameters than fixed beds</li> <li>Easy sorbent feeding and withdrawal</li> <li>Can control performance</li> <li>Uses smaller, more reactive sorbent</li> </ul>	<ul> <li>Bed mixing reduces breakthrough time for bed</li> <li>Increase gas bypassing</li> <li>Need bed support, inlet gas distribution, bed loading and unloading features and facilities</li> <li>Extensive particle attrition and elutriation</li> <li>Bed mixing reduces breakthrough time for bed</li> <li>Increase gas bypassing</li> <li>Need bed support, inlet gas distribution, bed loading and</li> </ul>
	<ul><li>particles</li><li>Results in smaller vessel diameters than fluidized beds</li></ul>	<ul> <li>unloading features and facilities</li> <li>Extensive particle attrition and elutriation</li> <li>Smaller vessel diameter than fluid bed, but much taller</li> </ul>
Filter- Reactor	<ul> <li>Uses very small sorbent particles, highly reactive, and potentially cheaper</li> <li>Ease of sorbent feeding and performance control</li> <li>Can feed multiple sorbents</li> <li>Inherent uniform coating of filter elements for uniform removal performance</li> <li>Inlet gas can contain significant particulate content</li> <li>Outlet gas will be particle-free</li> <li>Protects downstream equipment and can utilize or recover upstream particulate</li> <li>Can control pressure drop to maintain flow capacity</li> </ul>	<ul> <li>Relatively complex manifolding to hold filter elements</li> <li>Possible damage to filter elements, causing sorbent leaks</li> <li>Uncertain filter cake contaminant removal performance – reaction kinetics vs. mass transfer resistances for fine, packed particles</li> <li>Uncertain filter cake sintering</li> <li>Pulse cleaning may allow gas breakthrough momentarily</li> </ul>

 Table 2.1 – Characteristics of Gas-Sorbent Polishing Contactors

- The fixed bed reactor for large-scale gas flows is not a simple design, but requires means to uniformly distribute the inlet gas across the inlet side of the packed bed, and means to uniformly withdraw the gas from the fixed bed to ensure uniformity of flow. This is usually done with large distribution plates and layers of large pellet beds that also support the adsorbent bed, and result in increased pressure drop across the reactor vessel.
- The fixed bed reactor must be designed to maximize the sorbent bed capacity, so that the number of vessels and the frequency of switching is minimized, and to maintain acceptable vessel pressure drop. This is most economically accomplished in shop-fabricated vessels, limiting the maximum vessel outer diameter to about 4.0 m (13-ft).
- The fixed bed reactor is prone to plugging if the inlet gas contains any amount of entrained particulate, and even with almost particulate-free fuel gas will result in gradual increasing pressure drop due to corrosion products, chemical deposition, and settling of the fixed bed.
- The fixed bed reactor is a source of particle emission into the gas. Even with low gas velocities through the fixed bed, the bed particles are subject to high levels of crushing forces and locally high gas velocities near the gas distribution plates, as well as particle chemical decrepitation within the bed, and such particle emissions cannot be tolerated in the industrial application.

Most of the fixed bed issues are shared by the moving bed contactors, but some additional issues also arise, as are listed in Table 2.1. Moving beds are only selected for contaminant polishing where heat effects are small and the inlet gas is free of particulate. Fluid beds and transport beds result in high rates of gas and sorbent bed mixing that reduces reaction driving forces, but are advantageous if high heat generation rates exist to provide more uniform temperature conditions. They can also tolerate moderate particulate content in the inlet gas, although such particulate can plug or erode the inlet gas distribution equipment. The fluid bed and transport bed contactors are also subject to very high sorbent particle attrition and elutriation that require these contactors to be operated with specially-fabricated sorbent particles that are durable but also reactive in the contactor environment.

The proposed filter-reactor acts like a semi-continuous fixed bed reactor, with each filter cake section acting as a continuously-fed, fixed bed reactor that is periodically removed from service for an instant during pulse gas cleaning. The filter-reactor has no tendency for plugging at the temperature of operation selected and maintains particle-free outlet gas conditions. This evaluation projects the filter-reactor has a high potential to provide high levels of contaminant removal performance, potentially using cheap, fine, unsupported sorbent particles.

#### Filter-Reactor Novel Gas Cleaning Development Status

The gasification technology, the raw gas heat recovery technology, the air separation technology, the conventional gas cleaning technologies, the Methanol Synthesis technology, and the Power Island technology applied in this evaluation represent commercial technologies. The Filter-Reactor Novel Gas Cleaning processes described here are the only non-commercial technologies addressed. The individual components of the Filter-Reactor Novel Gas Cleaning processes are primarily commercial, or near-commercial components, or are currently being developed in various prototype development activities.

The most significant development aspect of the Filter-Reactor Novel Gas Cleaning process is the adaptation of barrier filter technology to combined use as a chemical reactor utilizing injected sorbent particles to remove gas-phase contaminants. The major equipment components of this polishing process, the barrier filter and sorbent handling and feeding equipment, have reached a mature status and have been demonstrated at large scales, but the use of barrier filters as gas-particle reactors has seen only limited testing (Newby et al., 1995). Field Testing of a barrier filter at the Tidd PFBC demonstration project has provided evidence of the effectiveness of the barrier filter as a filter-reactor, showing considerable removal of SO<sub>2</sub> from the process flue gas by reaction with entrained dolomite particles. Modeling of the filter-reactor suggests that effective contaminant control will result by 1) effective dilute-phase sorbent-

gas contacting with high internal recirculation at contact times of 1 to 10 seconds, and 2) dense-phase contacting through uniform, dense sorbent filter cakes, with gas residence times of 0.1 to 1 second.

The Filter-Reactor Novel Gas Cleaning process is in its early stages of development in regard to the selection of sorbents, the identification of acceptable operating conditions, and the establishment of the performance of barrier-filters for gas-phase contaminant removal. Previously reported laboratory testing has been used to select appropriate sorbents and to identify their probable operating temperatures in the Novel Gas Cleaning process as the starting point for this evaluation (Newby et al., 2001).

#### Barrier Filter Technology Status

Barrier filters are in use commercially in three operating IGCC power plants, the Buggenum IGCC plant (Eurlings and Ploeg, 1999), the Wabash River IGCC plant (Wabash River Energy Ltd., 2000), and the Puertollano IGCC plant (Mendez-Vigo et al., 1997) for high-temperature gas particulate removal prior to low-temperature, dry-gas cleaning, although the barrier filter operating temperatures do not exceed 371°C (700°F) in any of these applications. This efficient particle removal provides benefits to the downstream gas cleanup processing by eliminating particulate contamination of the condensate and solvent streams generated in the low-temperature cooling process and desulfurization process. The particle removal performance and operating reliability of these barrier-filters have been acceptable once their design features and operating procedures were optimized. While the commercial IGCC barrier filter experience has been limited to temperatures up to about 371°C (700°F), considerable test experience in coal fuel-gases and combustion-gases up to 927°C (1700°F) has been accumulated (Newby et al., 2001, Lippert et al., 2001). Ceramic filter elements and metal filter elements, in the form of "candles", typically 60 mm (2.4 inches) in diameter and with lengths of 1.5 m (59 inches), are the principle type of barrier filter elements of interest today.

Siemens barrier filter testing experience has shown that ceramic filter elements can be successfully operated at very high temperatures if the filter system design and operation protects the ceramic candles from thermal shock and mechanical vibration damage. Metal filter elements have lower temperature operating limits than ceramic filter elements and are subject to corrosion damage and pore plugging. Metal filter candles are also considerably more expensive than ceramic filter candles. Some filter ashes, if exposed to temperatures above a critical level can result in severe filter cake sintering, and "bridges" can result that upset the barrier-filter performance and operability. Highly reliable operation of barrier filters can be achieved with well-behaved filter ashes in fuel gases at temperatures up to about 650°C (1200°F).

#### Humid-Gas Cleaning Technology Status

Humid-gas cleaning processes (so-called "hot", or "warm" gas cleaning) are primarily founded on the principle of contacting the process gas with solid sorbent particles that react heterogeneously with the desired contaminant species for contaminant removal. The descriptor "hot" or "warm" is arbitrary and indicates that the temperature of the sorbent-gas contactor is far above the dew point of the gas being cleaned. In principle, the contactor temperature is selected to match the desired temperature of the gas in its process application, or is selected to yield a sufficiently high rate of reaction for the gas cleaning process to be economically acceptable. Again in principle, operation of the gas cleaning system at this higher temperature, which eliminates condensation from the gas and delivers cleaned gas at an elevated temperature, has the potential to result in higher power plant thermal efficiencies than using conventional, low-temperature, dry-gas cleaning. The promise of improved IGCC power plant thermal performance and cost by using humid-gas cleaning provides great incentive for developing such technology.

Humid-gas cleaning for IGCC application has been the subject of research and development for several decades, and numerous sorbent types, contactor types, and process configurations have been proposed, tested, and characterized, but without reaching commercialization (Parsons Power Group, 1997; Sierra Pacific Power Co., 1994; O'Hara et al, 1987; Cicero et al, 2003). The only example of a

"near commercial" humid-gas cleaning system for IGCC is the use of limestone within a fluidized bed gasifier, operating at about 871°C (1600°F) to remove as much as 95% of the coal sulfur content, and operating as a once-through sorbent process (KRW and GTI fluid bed gasifier). Such in-gasifier desulfurization only deals with sulfur removal and the IGCC gas still requires significant cleaning of several other contaminants before it can satisfy gas cleaning requirements.

The types of humid-gas cleaning processes that have been previously considered for IGCC are designated as "bulk" gas cleaning systems in this evaluation, in that they have the capability to remove a very large portion of the fuel gas contaminant (maybe 90-99%), but cannot achieve low enough contaminant concentration to satisfy the stringent gas cleaning requirement of many applications. Bulk gas cleaning systems have been proposed for sulfur species and halides to achieve moderately low contaminant levels, operating at temperatures up to about 650°C (1200°F). The bulk sulfur removal processes proposed are regenerative, generating an acid gas stream suitable for sulfur recovery, and recycling sorbent particles to the desulfurizer. The acid gas stream generated is usually in the form of a gas consisting primarily of SO<sub>2</sub> and N<sub>2</sub>, and such a gas requires reduction by a reducing agent if elemental sulfur is the desired sulfur recovery product. Alternatively, sulfuric acid, or a sulfur product such as gypsum could be made directly from the acid gas by contact with limestone, but would result in a large sulfur release in the tail gas (Wasaka and Suzuki, 2003).

Many bulk gas desulfurization sorbent types (for example, iron-, nickel-, manganese-, copper-, and zinc-based sorbent particles on various support structures) have been tested at laboratory scale. Zinc-based sorbents, operated at temperatures up to about 593°C (1100°F), have the highest thermodynamic potential for efficient sulfur removal and have reached the highest level of commercial-readiness. These sulfur sorbents have been considered for use in a variety of gas-particle contactor types: fixed beds, moving beds, fluidized beds and transport beds. Of these, the most developed and promising may be the transport bed. Various test and development efforts are currently underway using zinc-based sulfur sorbents in transport beds (Yi, et al, 2003; Everitt and Bissett, 2003; Silverman et al, 2003; Gangwal et al, 2003) and these provide the basis for the bulk desulfurization process step used in the Filter-Reactor Novel Gas Cleaning process in this evaluation.

Bulk halide removal has generally been based on the use of cheap, once-through sodium-based sorbents that are contacted with process gas in fixed beds or fluidized beds at temperatures as high as 816°C (1500°F). These have high performance capability and form the basis of the bulk halide removal process used in the Filter-Reactor Novel Gas Cleaning process in this evaluation.

Currently, proposed humid-gas cleaning processes do not address all of the gas contaminants that must be controlled in IGCC. There are no hot fuel gas sorbents for fuel-bound nitrogen species (e.g., ammonia and hydrogen cyanide) and most testing has been focused on catalysts capable of promoting the thermal decomposition of ammonia. Humid-gas cleaning processes depend on the condensation of many contaminant species to form liquid and solid phases at relatively high temperatures that can then be physically removed from the gas (e.g., alkali metal vapors and various other metal vapors). Other important contaminants, such as mercury and metal carbonyls, have seen little development effort for their removal under humid-gas cleaning conditions. Overall, there is currently no humid-gas cleaning process available that can, even in principle, meet all of the gas cleaning requirements dictated for IGCC power generating plants. The status of humid syngas cleaning for Chemical Synthesis applications, where syngas cleaning requirements are much more stringent, is even further from realization.

## 3. EVALUATION BASIS

A plant design basis has been established that allows direct comparison of the performance and cost of the conventional, dry-gas cleaning technology with the Filter-Reactor Novel Gas Cleaning process applied for meeting the gas cleaning requirements of IGCC power generation and IGCC-Methanol Synthesis. The level of detail defined for this conceptual evaluation by this basis is sufficient to clearly identify the potential, <u>relative merits</u> of the Filter-Reactor Novel Gas Cleaning technology.

## **3.1 PLANT DESIGN BASIS**

The major aspects of the design basis selected for the IGCC and Methanol Synthesis plants for this evaluation are listed below. In both applications, the plant is designed with sufficient fuel gas and syngas flow rates to match the requirements of the gas turbine combined-cycle, and the desired methanol production rate.

## **Power Island**

A gas turbine having characteristics representative of "F" gas turbine technology (for example, the Siemens SGT6-5000F gas turbine) is used in a single-train, combined-cycle Power Island configuration. The gas turbine air compressor and combustors are modified to handle the required air extraction, to accommodate IGCC low heating-value fuel gas operation, and to meet low-NOx emission requirements. It is assumed that a Selective Catalytic Reactor is not needed for the plant to achieve the required NOx emission limit. The turbine combustor air-side pressure drop is fixed at 60 kPa (8.7 psi) for all the evaluation cases. The gas turbine air compressor inlet loss is fixed at 1 kPa (0.14 psi), and the turbine exhaust pressure is set at 108 kPa (15.7 psia) in all cases. The turbine firing conditions are fixed at those used with natural gas operation, and the gas turbine cooling air rate and distribution to the turbine stages are comply with their normal, natural gas-fired turbine values for all of the evaluation cases.

## **Steam Bottoming-cycle Conditions**

The steam bottoming cycle uses a single-pressure, heat recovery steam generator (HRSG) system with reheat,

- superheat steam temperature 538°C (1000°F),
- reheat steam temperature 538°C (1000°F),
- steam throttle pressure 10.1 MPa (1465 psig),
- condenser pressure 7 kPa (0.98 psia).

## **Plant Nominal Capacity**

The IGCC power plant has a nominal, net generating capacity of about 300 MWe, resulting from the generating capacity of the Power Island gas turbine and steam-bottoming plant, and the net power losses associated with the gasification and gas cleaning processes. The Methanol Synthesis plant has a nominal, net generating capacity of about 300 MWe, with a selected production rate of methanol of about 218 tonne/day (240 tons/day). In both cases, the 300 MWe size represents the expected nominal capacity of "F" engine, combined-cycle technology.

## **Coal Characteristics**

A bituminous, high-sulfur coal, Illinois Number 6, is the plant fuel. Table 3.1 lists its moisture-free composition, heating value, and as-fed moisture content. Chlorine and mercury contents of the coal are included.

### **Coal Feed Rates**

The IGCC power plant, as-fed coal rate is 98,847 kg/hr (217,921 lb/hr) with energy input of 759 MW(t) (2,589 x  $10^{6}$  Btu/hr). The Methanol Synthesis coal feed rate is 115,058 kg/hr (253,661 lb/h) with energy input of 883 MW(t) (3,013 x  $10^{6}$  Btu/hr).

Constituent	Wt%
Н	5.32
0	10.0
С	69.4
Ν	1.25
S	3.85
Ash	10.0
Cl	0.18
Hg	6.3 X 10 <sup>-8</sup>
SUM	100
heating value (HHV, dry)	32.05
MJ/kg (Btu/lb)	(13,781)
moisture, as fed (wt%)	4.2

 Table 3.1 - Illinois Number 6 Coal Analysis (ultimate)

## **Plant Design Layout**

For this conceptual design evaluation, a single gasifier unit and a single gas cleaning train are used with no equipment spares, and are integrated with a single gas turbine combined-cycle Power Island, and a single Methanol Synthesis process. The air separation unit (ASU) and gas turbine air compressor are 100%-integrated, meaning that all of the air needed for the ASU is extracted from the turbine's air compressor. Actual commercial designs may call for 50%, or less air-side integration.

## **Coal Gasifier Type**

Gasifier technology representative of an oxygen-blown, entrained, single-stage, slagging gasifier is used. Coal is fed to this gasifier as a water slurry containing 69 wt% solids.

## **Gasifier Operating Pressure**

The IGCC gasifier operating pressure is selected to be sufficiently high, 2578 kPa (400 psia outlet), to deliver clean fuel gas to the gas turbine combustors with at least a 345 kPa (50 psi) pressure drop available for flow control, using moderate component design pressure drops in the process equipment. The Methanol Synthesis plant gasifier operating pressure is selected to be sufficient, 8791 kPa (1275 psia) outlet to deliver clean syngas to the Methanol Synthesis process at its inlet pressure of about 7033 kPa (1020 psia) – the high-pressure fuel gas stream is expanded and delivered to the gas turbine combustors with at least a 345 kPa (50 psi) pressure drop available for flow control.

## **Raw Gas Cooling Type**

Non-quench heat recovery is used to cool the raw gas, producing high-pressure, saturated steam.

#### **Ambient Conditions**

The plant site is assumed to be fixed at ISO-conditions, with ambient air at 15°C (59°F), 60% relative humidity, and a pressure of 101 kPa (14.7 psia). This evaluation is limited to a point-design, and the

influences of off-design ambient conditions on the equipment designs and process performance are not considered.

## **Cooling Water Conditions**

Cooling water is available at 21°C (70°F) with a supply pressure of 345 kPa (50 psia).

## **Ambient Air Composition**

The air composition (volume percent) used is

## Air Separation Unit Oxidant Composition

95% oxygen purity is specified with a composition (volume percent) of

 $\begin{array}{ccc} O_2: \ 95.0 \\ N_2: \ \ 4.5 \\ Ar: \ \ 0.5. \end{array}$ 

## Air Separation Unit Nitrogen Composition

Two types of nitrogen are produced by the ASU and used in the plant, a low-purity nitrogen, and a high-purity nitrogen:

low-purity nitrogen (volume percent):

 $\begin{array}{l} O_{2:} & 0.5 \\ N_{2:} & 98.5 \\ Ar: & 1.0. \end{array}$ 

high-purity nitrogen (volume percent):

 $\begin{array}{l} O_{2\,:} & 0.03 \\ N_{2\,:} & 99.92 \\ Ar : & 0.05. \end{array}$ 

Significantly different quantitative results from those produced in this evaluation would be expected if some of the major power plant design basis criteria were changed, using instead:

- alternative coals that are in wide use in the United States, such as Power River Basin sub-bituminous,
- alternative, commercial coal gasifier technologies, such as two-stage, oxygen-blown, entrained, slagging gasifiers, or fixed or fluidized bed gasifiers, or transport gasifiers,
- alternative raw gas cooling schemes, such as water quench, or recycle gas quench.

Nonetheless, it is expected that the same qualitative conclusions with respect to the potential benefits and issues for the Filter-Reactor Novel Gas Cleaning technologies would result.

# 3.2 GAS CLEANING AND CONDITIONING REQUIREMENTS

The gas cleaning process and related gas conditioning steps must meet certain specifications that address:

- the plant environmental emissions,
- the protection of the gas turbine and other Power Island components,
- the protection of the Methanol Synthesis reactor.

These specifications are of critical importance to the design of the gas cleaning processes, and achieving these specifications, while maintaining the overall plant performance and economics at acceptable levels, is a key measure of the success of the gas cleaning technology.

## **IGCC Gas Cleaning Specifications**

The IGCC power plant gas cleaning technology evaluation considers two sets of fuel gas cleaning requirements, designated "Current Standards" and "Future Standards", representing a major parameter in the assessment of the Filter-Reactor Novel Gas Cleaning technology. The fuel gas cleaning requirements for the Current Standards, and for the hypothesized Future Standards are listed in Table 3.2. These fuel gas cleaning requirements are developed from three sets of criteria:

- 1) representative, new coal-fired power plant environmental emission standards, that translate stack gas emission requirements into fuel gas cleaning requirements,
- 2) hypothetical, future environmental emission standards that will ensure that IGCC is environmentally competitive with natural gas-fired power plants,
- 3) representative gas turbine specifications for fuel gas contaminants.

Nine categories of fuel gas contaminants are listed in Table 3.2, and for each the maximum allowable content of the contaminant in the fuel gas is shown, along with an indication of the basis for the contaminant specification. Comments are also provided on each to provide additional perspective. The fuel gas cleaning requirements listed apply the following specific assumptions:

- the fuel gas location where the fuel gas cleaning criteria in Table 3.2 apply is in the moisture-free, clean fuel gas being fed to the turbine combustors, prior to nitrogen dilution,
- the clean, moisture-free fuel gas is assumed to have a heating value of about 200 Btu/sft<sup>3</sup>,
- the fuel gas cleaning criteria assume that <u>no</u> contaminants exist in the air, steam, or nitrogen streams that are fed to the turbine.

The IGCC power plant Current Standards are based on the "best-of-current-practice" reported for existing IGCC power plants. The following set of contaminant control targets result:

- SOx: minimum of 99% coal sulfur removal "net" for the entire power plant,
- NOx: 15 ppmv (corrected to 15% oxygen, dry) at the plant stack,
- particulate: 0.1 ppmw in the clean fuel gas,
- mercury: 90% coal mercury removal,
- Power Island contaminants in the fuel gas to meet gas turbine standards:
  - halides: 5 ppmv before turbine to protect low-temperature heat recovery in the power island,
  - metals (Ni, Fe carbonyls): no specific controls for metal carbonyls are included,
  - particulate: 0.1 ppmw in the fuel gas.

The IGCC power plant Future Standards, approaching the emissions performance of natural gasfired power plants, have the following set of targets:

- SOx: 99.98% coal sulfur removal "net" for entire power plant,
- NOx: 5 ppmv (corrected to 15% oxygen, dry) at the plant stack,
- particulate: 0.1 ppmw in the fuel gas,
- mercury: 95% coal mercury removal,
- Power Island contaminants:
  - halides: 5 ppmv before turbine to protect low-temperature heat recovery in the Power Island,
  - metals (Ni, Fe carbonyls): no specific controls for metal carbonyls are included,
  - particulate: 0.1 ppmw in the fuel gas.

The SOx, NOx, particulate, and mercury requirements represent the emissions from the entire power plant, the stack and all other exhaust vents in the plant. The Power Island contaminant control targets are the same for the Current Standards and the Future Standards. The Power Island contaminants

need only be controlled to protect the gas turbine and the downstream power island equipment. Alkali metals, and several other potential contaminants listed in Table 3.2 are expected to be inherently removed by condensation mechanisms in both the conventional gas cleaning and the Novel gas Cleaning processes.

Contaminant	Evaluation Environmental Emission Requirements	Power Island Fuel Gas Requirements (moisture-free fuel gas prior to nitrogen dilution)	Basis	Comments	
Total sulfur (H <sub>2</sub> S, COS, etc.)	Current: 99% total removal	750 ppmv	Current: best reported IGCC performance.	Emission requirement more stringent than turbine requirements.	
	Future: 99.98% total removal	750 ppmv	Future: competitive with emission from natural gas-fired plant		
Total halide (Cl, F, Br)	Current: none Current: 5 ppmv Protect gas cleaning	Protect gas cleaning sorbents/ minimize	Lower halide may be desirable to heat		
	Future: none	Future: 5 ppmv	power island low- temperature corrosion.	recovery protection during transient operations	
Particulate (char, sorbents, metal corrosion products)	Current: PM-10 standards	Current: 0.1 ppmw	Reflects best performance reported	More stringent than PM-10 and PM-2.5, or	
	Future: PM-2.5 standards	Future: 0.1 ppmw	for barrier filters.	turbine fuel gas standards of 0.7 ppmw	
Total fuel-nitrogen (NH <sub>3</sub> , HCN)	Current: NOx emission of 15 ppmv (15% O2, dry) Future: NOx emission of 5 ppmv (15% O2, dry)	Current: fuel bound nitrogen 225 ppmv for NOx control Future: FBN 375 ppmv for NOx control	Acceptable NOx generation in turbine combustors; 75% NH <sub>3</sub> catalytic decomposition in cleanup process	Assumed staged, or catalytic diffusion burners will limit fuel- nitrogen to 5% (current)/ 3% (future) conversion to NOx.	
Total mercury	Current: 90% removal	Current: no specific limit	Expected mercury environmental standards for coal-	No impact on gas turbine protection.	
	Future: 95% removal	Future: no specific limit	fueled power plants		
Trace elements (As, Se, etc)	No current standards	No standards apply		Mercury removal provides removal potential for these trace elements.	
Total alkali metals (Na, K, Li vapor and solid phases)	None	Current: 100 ppbv	Gas turbine corrosion and deposition standards	Removed efficiently by condensation in fuel gas	
		Future: 100 ppbv		cleaning system at 1000°F or less.	
Volatile metals (V, Ni, Fe, Pb, Ca, Ba, Mn, P)	None	Current: 10 -1000 ppbv Future: 10-1000 ppbv	Gas turbine corrosion and deposition	Metal carbonyls (40 ppbv required) are only issue in IGCC .	

Table 3.2 - IGCC Gas Cleaning Current and Future Standards

Additional protection against metal carbonyls and corrosion-derived particulate is not included in this evaluation. Low-temperature adsorption processes and a final filter stage can be added with dry gas cleaning to remove metal carbonyls (Collodi and Brkic, 2003), but metal carbonyl minimization and particulate protection are inherent aspects of the Filter-Reactor Novel Gas Cleaning technology.

Fuel-bound nitrogen species, such as ammonia and HCN, are not themselves contaminants in IGCC, but they are precursors to NOx generated in the gas turbine combustors. While the conventional, low-temperature gas cleaning technology provides effective removal of fuel-bound nitrogen species from the fuel gas, the Filter-Reactor Novel Gas Cleaning technology relies primarily on the use of advanced low-NOx combustors using staged combustion and possible catalytic combustion mechanisms for fuel-bound nitrogen NOx control.

The conventional gas cleaning technology and the Novel Gas Cleaning technology both generate solid wastes and process-condensate streams that need to be processed and controlled to minimize emissions and other environmental impacts. It is important to minimize the effects of both solid waste and liquid waste streams.

Representative fuel gas contaminant specifications for the gas turbine are listed in Table 3.3 for both natural gas and coal-gas-fired SGT6-5000F turbines. Conventional dry-gas cleaning technologies are expected to be able to satisfy these specifications, with the possible exception of iron and nickel, where volatile forms (carbonyls forms Ni(CO)<sub>4</sub> and Fe(CO)<sub>5</sub>) may be present in the fuel gas in some cases (Bonzani and Pollarolo, 2004).

Fuel Gas	Natural gas	IGCC fuel gas	Comments
Constituent	SGT6-5000F	SGT6-5000F	Comments
Constituent	Specification	Specification*	
Total Sulfur	0.5 wt%	750 ppmv	Relates to Power Island heat recovery protection this
$(H_2S, COS, etc.)$	0.0	, coppin,	specification far exceeds emission limits, so is not an issue.
Fuel-Nitrogen	0.015 wt%	40 ppmv	Relates to stack NOx emissions. Specification depends on type
(NH <sub>3</sub> , HCN, etc.)			of combustor.
Particulate	0.00015 lb/ x	0.7 ppmw	Relates to turbine erosion/deposition protection. Specification is
	$10^6$ Btu		more stringent than plant emission requirement for particulate.
Mercury	None	None	No impact on Power Island.
Trace Toxics	None	None	No impact on Power Island.
(As, Se, etc.)			-
Total alkali	0.5 ppmw	0.1 ppmv	Relates to turbine corrosion protection. Gas cleaning at
metals			temperatures < 1000°F should achieve this by condensation.
(Na, K, Li)			
Total Halogens	6.0 ppmw	5 ppmv	Relates to power island heat recovery protection during transient
(Cl, Br, F, I)			periods.
Volatile Metals			All relate to turbine deposition/corrosion protection
V	0.5 ppmw	0.05 ppmv	Should not be an issue with small V-content of most coals
Ca	10.0 ppmw	1.2 ppmv	Should be removed cleanup system by condensation
Ba	2.0 ppmw	0.1 ppmv	Should be removed cleanup system by condensation
Pb	0.5 ppmw	0.01 ppmv	Should be removed cleanup system by condensation
Mn	2.0 ppmw	0.2 ppmv	Should be removed cleanup system by condensation
Р	2.0 ppmw	0.3 ppmv	Should be removed cleanup system by condensation
Fe	0.5 ppmw	0.04 ppmv	Possible issue Fe-carbonyls form at temperatures < 400°F
Ni	0.5 ppmw	0.04 ppmv	Possible issue – Ni-carbonyls form at temperatures < 900°F

Table 3.3 – Gas Turbine Fuel Gas Contaminant Specifications

\* contaminant content in moisture-free syngas prior to humidification or nitrogen dilution for typical oxygen-blown gasification syngas

Metal carbonyls may be a greater problem with refinery residue gasification than with coal due to the high iron and nickel content of residues. Metal carbonyls, forming at temperatures less than 482°C (900°F) for Ni and 204°C (400°F) for Fe, decompose when raised above these temperatures, depositing

nickel and iron metals on available surfaces. These deposits can disrupt the operation of some gas cleaning stages in dry-gas cleaning (e.g., COS hydrolysis reactor, or water gas shift reactor), as well as degrade the performance of gas turbine combustors and other turbine hot parts (Bruijn et al, 2003). Metal carbonyls can also form through fuel gas contacting piping and equipment metal surfaces at appropriate temperatures. Metal carbonyls are expected to be less likely to form in a humid-gas cleaning process where Fe- and Ni-particulate is removed from the fuel gas before the temperature is lowered to the metal carbonyl formation temperature, and where contact with metal surfaces at temperatures lower than the formation temperature is minimized.

The IGCC power plant fuel gas cleaning process must also simultaneously meet other performance criteria:

- should not result in a decrease in coal-carbon utilization in the plant,
- have acceptable impact on plant solid waste (i.e., solid waste volume, cost of hazardous waste disposal),
- have acceptable water consumption and water discharge, removing absorbed halides and ammonia from condensate streams and disposing of these halide and ammonia streams with minimal environmental impact,
- provide fuel gas conditioning for acceptable NOx combustor emission (e.g., water vapor content and nitrogen dilution for acceptable peak flame combustion temperature),
- integrate with the power island for acceptable power plant thermal efficiency,
- the conditioned fuel gas reheat maximum allowable temperature is 371°C (700°F) based on current control valve materials, but temperatures up to 538°C (1000°F) are acceptable, although more expensive valve materials are needed.
- have acceptable gas cleaning train pressure drop,
- have acceptable gas cleaning train operability, reliability, and availability,
- have acceptable gas cleaning train capital investment, and operating & maintenance cost.

The last three items – pressure drop, operating reliability, and cost -- are design-performance tradeoff parameters that interact with each other but do not have specific numerical targets.

## Methanol Synthesis Gas Cleaning Requirements

The gas cleaning requirements considered for Methanol Synthesis are broken into three classes:

## **Environmental Air Emissions**

- SOx: 99.98% sulfur removal "net"
- NOx: 5 ppmv (corrected to 15% oxygen, dry)
- mercury: 95% removal

# **Power Island Fuel Gas Contaminant Standards**

- halides: 5 ppmv in the fuel gas (dry)
- fuel-nitrogen (NH<sub>3</sub> + HCN): 225 ppmv (dry)
- metal carbonyls: 40 ppbv in fuel gas (dry)
- particulate: 0.1 ppmw

# **Methanol Syngas Requirements**

- total sulfur: 60 ppbv (dry)
- total halides: 10 ppbv (dry)
- NH<sub>3</sub>: 10 ppmv (dry)
- HCN: 10 ppbv (dry)
- metal carbonyls: 100 ppbv (dry)
- mercury: 95% removal
- particulate: 0.1 ppmw

The environmental standards applied are identical to the Future Standards case used for IGCC. The Power Island standards are the same as those used for IGCC. The Methanol Synthesis standards for syngas are extremely stringent and are based on protecting the Methanol Synthesis catalyst.

# 3.3 MATERIAL & ENERGY BALANCE ESTIMATION AND EQUIPMENT SIZING

Process flow sheet development and materials & energy balance computations were performed using the ChemCad process simulator software (by Chemstations, Inc., Houston, Texas) for both the conventional, low-temperature gas cleaning processes and for the Filter-Reactor Novel Gas Cleaning processes. Considerable effort was applied to properly converge the process material balances due to the low concentrations of several of the chemical contaminants of interest in the evaluations. Material & energy balances are highly dependent on process reactor conversion efficiency assumptions and separation equipment contacting efficiency assumptions, and these assumptions are listed with the process and equipment descriptions provided in the following sections of the report. For the Filter-Reactor Novel Gas Cleaning processes, several assumptions of sorbent compositions, feed rates, and reaction conversions were made based on available literature information and laboratory test results. These assumptions are listed in the descriptions of the processes and equipment in the report sections that follow.

Equipment and process sub-systems have been conceptually designed and specified using standard industrial sizing criteria and design factor estimates. For example, available industrial experience information and design criteria were input for:

- heat transfer coefficients, with fouling,
- heat exchanger pressure drops,
- heat exchanger minimum temperature approaches,
- reactor operating temperatures and pressure drops,
- reactor equilibrium temperature approaches,
- reactor residence times,
- compressor-type selection and efficiencies,
- pump-type selection and efficiencies,
- absorber and stripper column contacting efficiencies and pressure drops.

The representative SGT6-5000F gas turbine expander was scaled in each of the IGCC and Methanol Synthesis cases to yield the required compressor air delivery pressure and fuel gas delivery pressure, and to determine the air compressor pressure ratio and adiabatic efficiency. It is assumed that an adapted gas turbine air compressor would be utilized in each case, modifying the standard air compressor, with the compressor air flow rate adjusted to allow the same rotor inlet temperature as the standard, natural gas-fired turbine. It is assumed that the cooling air rate and its distribution to the vanes and rotors of the four stages of the turbine expander are the same in all of the cases and identical to that for the standard natural gas-fired turbine.

The ChemCad process simulator used in the process evaluation includes facilities for the design and specification of many of the equipment components and does rigorous design of heat exchangers, and absorption and stripping columns. Barrier filters and filter-reactors have been sized and designed using Siemens proprietary design codes. Heat exchanger designs have accounted for appropriate materials of construction and maintenance considerations.

Solids handling equipment are particularly important for the Filter-Reactor Novel Gas Cleaning technology. Equipment for on-site storage, transport, pressurization, and feeding of sorbents have been sized using typical specifications for vessel storage and holding times, transport gas requirements, and

vent gas handling. Likewise, equipment for waste solids cooling, depressurization, transport and storage have been designed by similar criteria. All solids handling equipment has been designed using a 50% excess capacity factor.

# 3.4 ECONOMIC CRITERIA AND COST ESTIMATION APPROACH

This is a conceptual design evaluation and the equipment costs and operating costs generated are approximations based on the use of purchased-equipment general cost correlations and operating cost factors extracted from prior IGCC plant cost studies. The objective of the cost estimates generated is to compare the relative cost potential and cost sensitivities of the conventional dry-gas cleaning technologies and the Filter-Reactor Novel Gas Cleaning technologies.

A "factored" cost evaluation, estimating the purchased and installed costs of the major equipment in the Gas Cleaning Sections of each plant has been performed. These cost estimates were generated by sizing all of the major gas cleaning equipment, using generalized equipment cost correlations and other available cost data to estimate the purchase price of each major equipment component, and applying "installation factors" to each item to estimate the installed equipment cost, or Base Erected Cost for the gas cleaning process. In some cases, for very expensive process components, vendor budgetary cost inputs have been obtained. The equipment costs of barrier filters have been developed using Siemens internal cost data and correlations.

The "balance-of-total plant", that is, the remaining plant apart from the Gas Cleaning Sections, has been assumed to have a cost nearly identical for all of the IGCC cases, being essentially unchanged in the cases. This "balance of total plant cost" has been estimated from previous conventional IGCC power plant cost studies that provide Base Plant Total Capital Requirement (TCR) values.

No process contingencies or project contingencies have been assigned for the Conventional or Filter-Reactor Novel Gas Cleaning processes. The Total Capital Requirement (TCR) is estimated from the values of the installed major equipment costs, using assumptions and procedures representative of the EPRI Technical Assessment Guide (EPRI, 1986). This results in the use of a constant ratio of Total Capital Requirement (TCR)-to-Bare Erected Cost equal to 1.5 applied in the estimates. The major cost assumptions applied, based on the general findings of numerous previous IGCC evaluations, were:

- Cost-basis year: end-2002
- Plant capacity factor: 65% for IGCC and 80% for Methanol Synthesis co-production plant
- TCR/Bare Erected Cost ratio: 1.50
- Base Conventional IGCC plant TCR: 1500 \$/kW
- Base Conventional Methanol Synthesis plant TCR: 1790 \$/kW

The balance-of-total plant cost was assumed the same for all of the plants, except for that of the steambottoming plant cost which was scaled, since its capacity changed significantly between the various plant cases.

The cost-of-electricity (COE) was estimated on a "first-year of operation" basis. It was estimated using a Capital Charge of 15% per year, and Operating and Maintenance (O&M) costs estimated, based on general results from numerous prior IGCC evaluations, as:

- Operating labor: 0.87% of TCR/yr
- Maintenance labor: 0.67% of TCR/yr
- Maintenance materials: 1.01% of TCR/yr
- Administrative and support labor: 0.39% of TCR/yr

The Fixed O&M cost is the sum of the operating labor, the maintenance labor and the administrative & support labor. The Variable O&M is equal to the maintenance materials. The Total O&M is the sum of the fixed and variable O&M values.

Process consumable costs, by-product values, and solid waste disposal costs have been assigned to the various import stream catalysts, chemicals and sorbents, and fuels consumed, and export streams produced by the process. The following cost assumptions were made:

# fuels: coal: 2.00 \$/10<sup>6</sup> Btu (HHV),

### chemicals, catalysts, and conventional sorbents:

- raw water: 0.04/kW-hr.
- Claus catalyst, hydrolysis catalysts, methanol synthesis catalyst makeup costs were neglected,
- methanol solvent for Rectisol process: 0.40 \$/lb,
- MDEA solvent: 1.00 \$/lb,
- Sulfur sorbent pellets for conventional Methanol Synthesis guard bed: 4.0 \$/lb
- mercury sorbent (sulfur-impregnated activated carbon) for conventional cleaning: 6.5 \$/lb,

# Novel Gas Cleaning sorbents:

- bulk halide sorbent: 0.03 \$/lb,
- bulk desulfurization sorbent: 3.0 \$/lb,
- polishing sulfur sorbent: 1.0 \$/lb,
- mercury sorbent: 3.0 \$/lb.

### by-products:

- slag by-product value: 0 \$/ton,
- sulfur by-product value: 50 \$/ton,
- methanol product value: 0.15 \$/lb

### disposal:

- ash and inert sorbent waste non-hazardous disposal: 20 \$/ton,
- mercury, once-through sorbent, hazardous disposal: 200 \$/ton.

The sorbent costs listed above are representative of expected, delivered prices for these sorbent materials as mature, commercial commodities. The mercury sorbent cost for the Filter-Reactor Novel Gas Cleaning technology is a hypothetical value since the actual sorbent has not yet been commercialized. The two sulfur sorbents for the Filter-Reactor Novel Gas Cleaning technology also have uncertain costs, and it is likely that even lower costs may result for these. Some of the chemicals and by-products listed above, such as methanol and sulfur, have unstable costs. All of the items listed can have significant variations in cost subject to the power plant location and market factors.

# 4. IGCC WITH CONVENTIONAL GAS CLEANING

An overall process flow diagram for IGCC with conventional dry-gas cleaning is shown in Figure 4.1. It portrays the relations between the major process systems in the plant and shows only the major process streams.

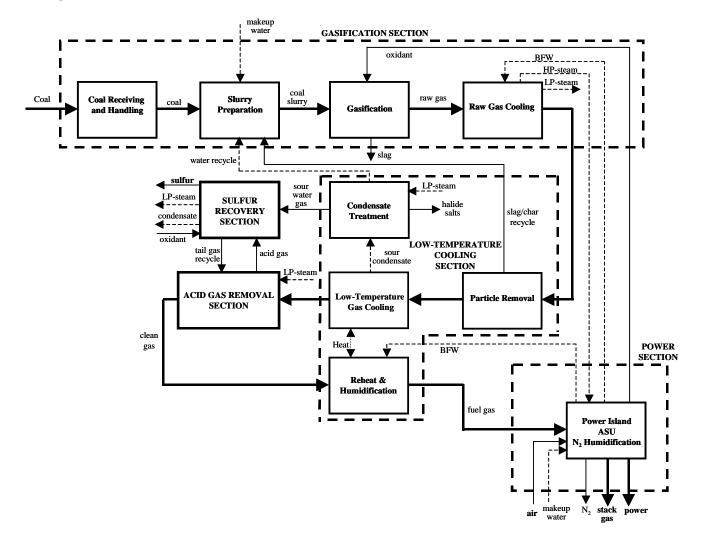


Figure 4.1 - Overall IGCC Plant with Conventional Dry-gas Cleaning

The overall process schematic is broken into five major plant sections: the Gasification Section, consisting of coal receiving and handling, slurry preparation, gasification, slag handling, and raw gas cooling sub-sections; the Low-temperature Cooling Section, the Acid Gas Removal (AGR) Section, the Sulfur Recovery Section, and the Power Section, consisting of the power island and air separation sub-sections. The diagram is highly simplified, not indicating all of the numerous sub-functions of each section and the numerous streams that pass between the various sections. The Low-temperature Cooling Section produces a large recycle water stream sent to the slurry preparation section, a sour-water gas stream sent to Sulfur Recovery, and a halide salts disposal product.

The major functions of these plant sections are:

Gasification Section (Figure 4.2)

- prepare coal slurry
- accept recycled slag
- accept recycled water
- accept recycled sulfur recovery tail gas
- gasify coal slurry
- cool raw fuel gas, generating saturated, HP-steam
- cool and separate slag from water for disposal

Low-Temperature Cooling Section (Figure 4.3)

- remove raw gas particulate (slag) and recycle to Gasification Section
- cool gas and remove condensate
- remove halide and ammonia by scrubbing with condensate
- perform COS and HCN hydrolysis
- extract soot blower gas for compression to Gasification Section
- remove mercury
- humidify and reheat clean fuel gas (from AGR Section)
- remove halide salts for condensate (Condensate Treatment), and water recycle to Gasification Section
- strip ammonia from condensate, generating sour-water gas sent to the Sulfur Recovery Section, and water recycle to Gasification Section

AGR Section (Figure 4.4 and Figure 4.7)

- desulfurize fuel gas
- produce acid gas suitable for sulfur recovery
- Sulfur Recovery Section (Figure 4.5)
- accept recycled sour-water gas
- oxidize acid gas with oxygen to generate desired H<sub>2</sub>S-SO<sub>2</sub> gas mixture
- decompose sour-water gas ammonia
- produce elemental sulfur product
- recycle tail gas to Gasification Section
- condensate recycle to Gasification Section
- produce IP-steam and LP-steam

Power Section (Figure 4.6)

- compress air for air separation and fuel gas combustion
- separate air to generate gasifier oxidant, sulfur recovery oxidant, and nitrogen streams
- humidify low-purity nitrogen stream and mix with clean fuel gas
- combust fuel gas for low-NOx production and gas turbine expansion for power generation
- recover heat from gas turbine exhaust gas for steam cycle power generation
- circulate BFW to Gasification Section and Low-Temperature Cooling Section

Material & energy balances have been developed for the five sections in sufficient detail to relate the dry-gas cleaning technology to its impacts on the power plant performance, and to provide a basis for sizing the associated major process equipment for cost estimation purposes. Only those aspects of the plant have been incorporated into the evaluation material & energy balances that are direct influenced by the gas cleaning functions.

# 4.1 IGCC WITH CONVENTIONAL GAS CLEANING TO MEET CURRENT STANDARDS

The case of conventional, low-temperature, dry-gas cleaning to meet Current Standards is considered in this section. Descriptions of the process sections of the IGCC power plant that are impacted

by the gas cleaning functions are provided, along with the characterization of major process streams and equipment.

#### **Gasification Section**

Figure 4.2 shows the process flow diagram constructed for the Gasification Section, and includes the Coal Receiving and Handling System and the Slurry Preparation System. Table 4.1 lists the characteristics of the major streams in this section. An entrained, oxygen-blown, single-stage, slagging gasifier, with coal-slurry feeding is used (Item 3). The coal is pumped (Item 1) as a slurry containing about 31wt% water, and this slurry is preheated to 149°C (300°F)(Item 2). Most of the slurry water is recycled water (treated condensate) from the Low-Temperature Cooling Section of the plant. Oxidant from the Power Section's Air Separation Unit combines with the coal slurry to generate high-temperature, raw gas and slag streams. An additional 1,346 kg/hr (2,968 lb/hr) of oxidant is generated for use in the Sulfur Recovery Section. The raw gas is cooled in a radiant cooler (Item 4), raising saturated, highpressure (HP) steam, and cooling the raw gas to about 824°C (1516°F) to solidify slag particles before cooling the gas further in a convective cooler (Item 7) to generate additional saturated HP-steam. The raw gas stream has heat content of 1,958 x 10<sup>6</sup> kJ/hr (1,856 x 10<sup>6</sup> Btu/hr) (LHV) and heating value of about 7.6 MJ/Nm<sup>3</sup> (194 Btu/scf), and includes about 9,979 kg/hr (22,000 lb/hr) of recycled, clean fuel gas used as soot blower gas in the heat exchangers. These values differ slightly in the two conventional fuel gas cleaning cases (Current Standards and Future Standards) due to the recycle of tail gas from the Sulfur Recovery Section to the gasifier in these cases. Net carbon loss from the gasification process is assumed to be 1 percent of the coal carbon content and is contained in the plant's slag waste stream.

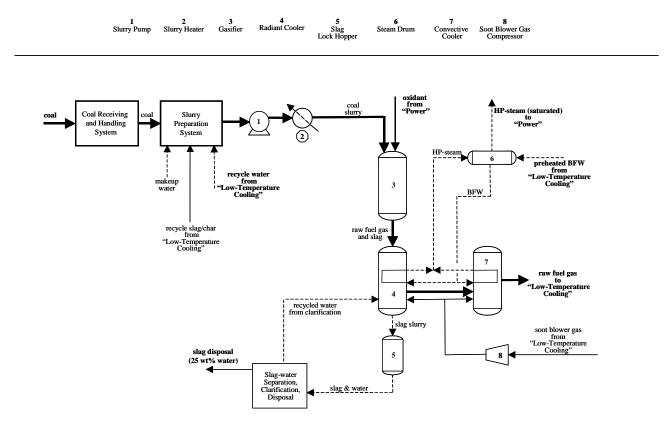


Figure 4.2 - Gasification Section for Conventional Dry-gas Cleaning

The fuel gas exits the Gasifier at about 1410°C (2570°F) and 2758 kPa (400 psia), and is 371°C (700°F) at the exit of the Convective Cooler, with the estimated composition listed in Table 4.2, not including entrained slag.

Stream name	Coal feed	Total slurry	Gasifier	Raw fuel	Slag	HP-steam
		water feed	oxidant	gas		
Molar flow, kmole/hr	8,810	2,429	2,899	11,448	224	14,767
Mass flow, kg/hr	98,847	43,767	92,357	248,353	10,511	266,032
Volumetric flow, m <sup>3</sup> /hr	122	45	2,377	23,324	13	4,900
Temperature, °C	26	56	81	371	667	318
Pressure, kPa	101	110	3585	2634	2468.	10,583
Enthalpy MJ/hr	-1.25E+05	-6.89E+05	4,283	-1.49E+06	-41,751	-3.52E+06
Molecular wt	11.2	18.0	31.9	21.7	46.9	18.0

Table 4.1 – Stream Characteristics for Gasification Section - Current Standards

Table 4.2 - Raw Gas Composition – Conventional Gas Cleaning with Current Standards

	Major constituents (vol%)
H <sub>2</sub>	23.63
CH <sub>4</sub>	0.30
СО	40.95
$CO_2$	10.93
H <sub>2</sub> O	21.30
$N_2$	1.57
Ar	0.14
Total	98.86
	Major contaminants (ppmv)
$H_2S$	9,524
COS	721
$CS_2$	0.7
S <sub>X</sub>	9
SO <sub>2</sub>	4
NH <sub>3</sub>	675
HCN	19
HC1	425
Hg (ppbv)	3

Gasification reaction conversions are estimated from thermodynamic equilibrium calculation, with suitable adjustments to methane, ammonia, and hydrogen cyanide to be representative of reported gasifier performance. The estimated distribution of contaminants in the raw gas issued from the gasifier is based on the empirical assumptions listed in Table 4.3. Significant sulfur content is assumed to remain in the gasifier slag, and the hot gas from the gasifier is assumed to be at equilibrium with respect to sulfur species. All of the coal's chlorine content is assumed converted to HCl in the raw gas, and other halides have been neglected in the evaluation. All of the coal's mercury is assumed to be issued in the raw gas as elemental mercury. Empirical conversions are assumed for ammonia and hydrogen cyanide. Metal carbonyls may be formed in the raw gas only at temperatures less than about 480°C (900°F) though gassolid reactions with the ash metal constituents, or with the materials of construction, and the level of formation is highly uncertain. There are a multitude of other trace species contained within the raw gas

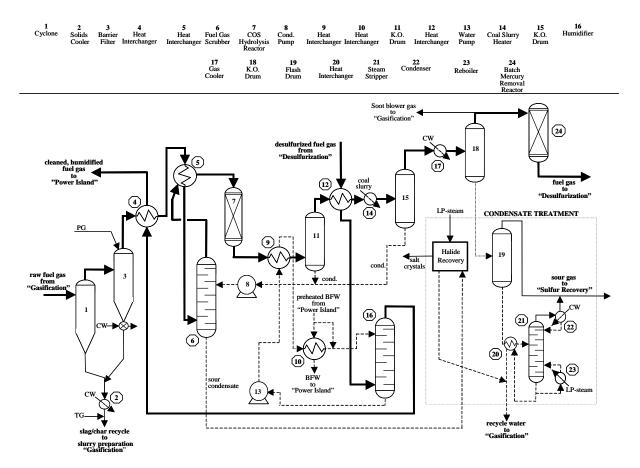
that are not listed and these are not expected to be of concern to either gas turbine protection or environmental emissions due to their expected condensed-form fate in the cleanup processes, or due to their low level of content in the fuel gas. The hot gas composition is assumed to be frozen at this level once it has passed through the raw gas cooler heat exchangers.

Contaminant	Generation	Contaminant forms
	(% of coal constituent)	
Sulfur species	90% (10% retained with slag)	$H_2S$ , COS, CS <sub>2</sub> , SO <sub>2</sub> based on
		equilibrium at exit temperature
Halides	100% of coal Cl content	HCl (other halides neglected)
Ammonia	25 % of coal nitrogen	NH <sub>3</sub>
Hydrogen cyanide	0.5 % of coal nitrogen	HCN
Mercury	100% of coal mercury content	Hg <sup>°</sup> only
Metal carbonyls	0 (generated <900°F only)	Fe(CO) <sub>5</sub> , Ni(CO) <sub>4</sub>

Table 4.3 – Distribution of Contaminants in Gasification Section Raw Gas

# **Low-Temperature Cooling Section**

Figure 4.3 represents the process flow diagram for the Low-Temperature Cooling Section used with conventional dry-gas cleaning. It includes all of the equipment treating the gas before it goes to the





AGR Section, as well as equipment for humidifying and reheating the cleaned fuel gas before it goes to the Power Section. Table 4.4 lists characteristics of major streams in the Low-Temperature Cooling Section. The raw gas from the Gasification Section is first pre-cleaned of entrained slag particulate using a conventional cyclone (Item 1). The subsequent sequence of processing steps is described below.

Stream name	Cooled raw gas	Slag/char recycle	Fuel gas to Desulfurization	Desulfurized fuel gas	Reheated fuel gas to Power Island
Molar flow, kmole/hr	11,448	43	8,549	8,190	10,166
Mass flow, kg/hr	248,353	2,562	192,203	177,397	213,005
Volumetric flow, m <sup>3</sup> /hr	23,324	2.0	9,933	9,802	22,934
Temperature, °C	371	371	38	44	310
Pressure, kPa	26,342	2,468	2,220	2,206	2,158
Enthalpy, MJ/hr	-1.49E+06	-12,177	-9.69E+05	-8.67E+05	-1.26E+06
Molecular wt	21.7	46.9	22.5	21.7	21.0

 Table 4.4 – Stream Characteristics for Low-Temperature Cooling Section

<u>Barrier filter</u>: A ceramic, or metal, candle-type barrier filter (Item 3) operates at about  $370^{\circ}$ C ( $700^{\circ}$ F) to removal particulate (solidified slag particles and char) from the raw gas to a level of < 0.1 ppmv as the first step in the cleaning process. The filter is pulse cleaned using compressed, high-purity nitrogen from the air separation unit. The collected particulate is combined with the cyclone catch and is pneumatically conveyed back to the gasifier for additional carbon conversion and slag collection, using recycled, clean fuel gas as the transport gas.

<u>Fuel gas coolers</u>: A process heat interchanger (Item 4), a shell-and-tube unit, is now used to cool the gas to about  $228^{\circ}$ C (443°F), while reheating the clean, humidified fuel gas to about  $310^{\circ}$ C (590°F) before it goes to the gas turbine combustors. This cooled gas then passes through a second heat interchanger (Item 5) that cools it to about 199°C (390°F).

<u>Fuel gas condensate scrubber</u>: The gas is next scrubbed in a bubble-cap, tray column (Item 6) with collected process condensate, about 22.435 kg/hr (49,460 lb/hr), to remove halides to a very low level, and results in the further cooling of the gas to about 158°C (316°F). The scrubber condensate is collected and treated in a crystallization process (Condensate Treatment) to remove the collected halides, primarily HCl, as ammonium chloride salts for disposal. The treated water is recycled to the Gasification Section's slurry preparation system.

<u>Gas reheater</u>: The gas is now reheated in a gas heat interchanger (Item 5) to about  $186^{\circ}C$  ( $366^{\circ}F$ ) in preparation for COS hydrolysis. This preheating step is required to elevate the temperature of the water-saturated gas at least  $28^{\circ}C$  ( $50^{\circ}F$ ) to protect the hydrolysis catalyst bed from possible condensate contact.

<u>COS hydrolysis</u>: The reheated gas is treated in a catalytic reactor (Item 7) to hydrolyze its COS content to  $H_2S$ , allowing more efficient sulfur removal to be performed downstream. This unit also effectively hydrolyzes HCN to ammonia.

<u>Fuel gas cooling and ammonia removal</u>: A series of process heat interchangers and water-cooled heat exchangers (Items 9, 12, 14, and 17) are used to cool the gas to about 38°C (100°F), simultaneously removing most of its water content and collecting process condensate that contains most of the gas ammonia as well as a portion of the sulfur species. The cooling water use represents the greatest thermal energy loss in the IGCC process. The condensate is collected and treated in a steam stripping process (Items 18, 19, 20, 21, and 22) to remove ammonia and sulfur gases. The treated condensate is recycled to the Gasifier Section to be used for coal slurry feeding. The stream of released sour-water gases from condensate processing is sent to the Sulfur Recovery Section. Its composition is listed in Table 4.5.

	Constituents (vol%)
$H_2$	1.16
$CH_4$	0.03
CO	2.18
$CO_2$	17.79
H <sub>2</sub> O	47.90
$N_2$	0.06
$H_2S$	8.03
COS	0.04
NH <sub>3</sub>	22.80
HCN	0.00
Total	99.99

 Table 4.5 - Sour-water Gas Composition - Current Standards

<u>Soot-blower gas</u>: A portion of the partially cleaned gas is withdrawn at this point in the process to be compressed and used as soot-blower gas in the raw gas heat recovery units, and as transport gas to carry the cyclone and filtered slag back to the gasifier.

<u>Condensate Treatment</u>: The halides are converted to ammonia salts for disposal, and ammonia is sent to the Sulfur Recovery Section to be decomposed in the high temperature Claus furnace. Considerable LP-steam is utilized in the condensate treatment process.

<u>Mercury removal</u>: The gas is now treated in a fixed bed reactor (Item 24) containing sulfur-impregnated, activated carbon to remove 90% of the gas mercury. This activated carbon bed is periodically drained and refilled with fresh adsorbent, and the used adsorbent is disposed as a hazardous material. This is assumed to be a single, fixed bed adsorption reactor operated in batch-mode. It is operated with no parallel-reactor switching, and is drained and refilled during annual power plant outage periods. This single vessel system is subject to the possibilities of bed flow bypassing, packed bed pressure drop increases, and elutriation of fine particulate. The criteria in the Eastman Chemical Synthesis plant for change out of their mercury sorbent bed is its increase in pressure drop beyond an established limit (Trapp et al., 2004). The composition of the gas sent to the AGR Section of the plant is listed in Table 4.6.

	Major constituents (vol%)	
$H_2$	29.96	
$CH_4$	0.39	
CO	51.93	
$CO_2$	13.97	
H <sub>2</sub> O	0.28	
$N_2$	2.00	
Ar	0.17	
Total	98.70	
	Major contaminants (ppmv)	
$H_2S$	12,955	
COS	2.5	
NH <sub>3</sub>	0.5	
HCN	0.004	
Hg (ppbv)	0.25	

 Table 4.6 - Gas Composition to AGR Section - Current Standards

The fates of the gas contaminants in the raw gas entering this section as estimated in the process simulation are listed in Table 4.7. The halides, ammonia, and hydrogen cyanide contaminants are very effectively removed from the raw gas, and are recovered from the condensate streams for final processing.

Contaminant	Removal	Contaminant conversions
	(% of inlet to Section)	
Sulfur species	5.3	Hydrolysis
		Partial condensate absorption
Halides	100	Condensate absorption
		Salt crystallization
Ammonia	99.95	Hydrolysis
		Condensate absorption
		Salt crystallization
Hydrogen cyanide	100	Hydrolysis
Mercury	90	Sorbent fixation

 Table 4.7 – Fate of Contaminants in Low-temperature Cooling Section – Current Standards

<u>Humidification</u>: The clean and dry fuel gas from the AGR Section is reheated to about 141°C (286°F), interchanging heat (Item 12) with the previous gas cooling-condensation process streams. This fuel gas is passed through a packed column (Item 16) where it is contacted with circulating boiler feed water to humidify the dry fuel gas to a water content of about 20 vol%. Heat for this humidification process is provided by some of the low-grade sources in the previous gas cooling steps. The humidification step is needed to produce a fuel gas that will have an acceptably low, peak flame temperature in the turbine combustors to minimize NOx formation.

<u>Fuel gas reheat</u>: The cleaned and humidified fuel gas is now reheated to about  $310^{\circ}$ C (590°F) in a heat interchanger (Item 4). There is no positive protection provided against metal carbonyls or corrosion particulate possibly generated in the low-temperature equipment. This reheated fuel gas is mixed with a humidified nitrogen stream from the air separation unit before being distributed to the turbine combustors. These combustors are diffusion flame burners, specifically designed for low heating-value fuel gas, and the fuel gas has been conditioned by humidification and nitrogen dilution so that the NOx generated will be less than 15 ppmv (corrected to 15 vol% O<sub>2</sub>, dry).

# **AGR Section**

Figure 4.4 represents the process flow diagram for the AGR Section to achieve the Current Standards requirements. Table 4.8 lists characteristics of major streams in the AGR Section. The cooled gas from the Low-Temperature Cooling Section is desulfurized in a conventional, amine-based absorption-stripping process. The gas passes through an amine absorption, bubble-cap tray column (Item 1), and the desulfurized fuel gas meets the requirement for an overall 99% sulfur removal performance in the power plant, since the plant is designed with no other sulfur release points in the power plant. Makeup water, at 274 kg/hr (603 lb/hr) and makeup MDEA solvent, at 34 kg/hr (75 lb/hr) are fed to the process. The lean solvent from the absorber is flashed (Item 5) to low pressure (about 30 psia), and flows through a stripping column (Item 9). Significant LP-steam, at 12 MW(t) ( $42 \times 10^6$  Btu/hr), is used to operate the solvent stripper reboiler (Item 10), generating a low-pressure, acid gas that is sent to a Sulfur Recovery Section. The flash gas is compressed (Item 3), cooled (Item 2) and circulated back to the inlet of the absorber column. A solvent reclaimer system is also needed to renew the MDEA solvent because of contaminant interactions.

Table 4.9 lists the composition of the desulfurized fuel gas. Table 4.10 lists the composition of the acid gas sent to the Sulfur Recovery Section.

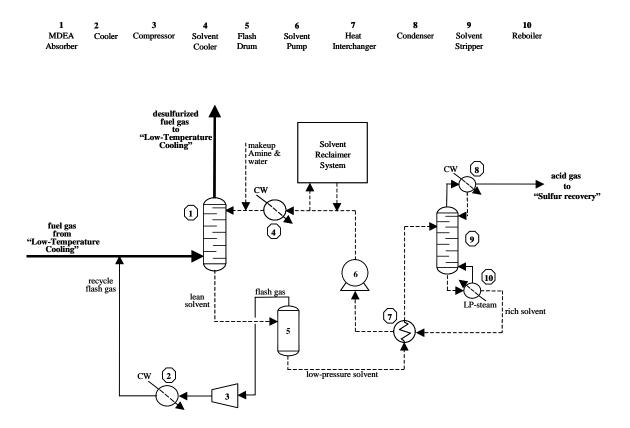


Figure 4.4 - AGR Section - MDEA for Current Standards

Stream name	Fuel gas from	Desulfurized	Acid Gas to
	LT-Cooling	fuel gas	Sulfur Recovery
Molar flow, kmole/hr	8,549	8,190	375
Mass flow, kg/hr	192,203	177,397	15,076
Volumetric flow, m <sup>3</sup> /hr	9,933	9,802	5,156
Temperature, °C	38	44	38
Pressure, kPa	2,220	2,206	186
Enthalpy, MJ/hr	-9.69E+05	-8.67E+05	-1.04E+05
Molecular wt	22.5	21.7	40.2

 Table 4.8 – Stream Characteristics for AGR Section – Current Standards

	Major constituents (vol%)	
H <sub>2</sub>	31.28	
CH <sub>4</sub>	0.40	
СО	54.21	
$CO_2$	11.50	
H <sub>2</sub> O	0.33	
N <sub>2</sub>	2.08	
Ar	0.18	
Total	99.98	
	Major contaminants (ppmv)	
$H_2S$	126.1	
COS	1.2	
NH <sub>3</sub>	0.5	
HCN	0.0	
Hg	0.0002	

 Table 4.9 - Desulfurized Fuel Gas Composition - Current Standards

 Table 4.10 - Acid Gas Composition - Current Standards

	Major constituents (vol%)
$H_2$	0.00
CO	0.01
CO <sub>2</sub>	67.35
H <sub>2</sub> O	3.35
$H_2S$	29.28
HCN	0.00
Total	99.99

Table 4.11 summarizes the fates of the contaminants within the desulfurization process.

 Table 4.11 – Fate of Contaminants in the MDEA AGR Section

Contaminant	Removal (% of inlet to Section)	Contaminant conversions
sulfur species	99.06	Solvent absorption
halides	NA	
ammonia	9.8	Solvent absorption
hydrogen cyanide	96.6	Solvent absorption
mercury	0	

The clean and humidified fuel gas composition after passing through the Low-Temperature Cooling Section is listed in Table 4.12.

	Major constituents (vol%)	
H <sub>2</sub>	25.20	
CH <sub>4</sub>	0.33	
CO	43.67	
$CO_2$	9.27	
H <sub>2</sub> O	19.70	
N <sub>2</sub>	1.68	
Ar	0.14	
Total	99.99	
	Major contaminants (ppmv)	
$H_2S$	101.6	
COS	0.1	
NH <sub>3</sub>	0.4	
HCN	0.0	
Hg (ppbv)	0.29	

 Table 4.12 - Cleaned and Humidified Fuel Gas Composition - Current Standards

### **Sulfur Recovery Section**

Figure 4.5 shows the process flow diagram for the Sulfur Recovery Section of the IGCC power plant. Table 4.13 lists characteristics of major streams in the Sulfur Recovery Section. The low-pressure, acid gas combined with waste gas from the sour-water condensate treatment process are treated in a Claus-type sulfur recovery process. The sour-water gas, containing significant ammonia and H<sub>2</sub>S, and an appropriate portion of the acid gas are combined and are burned in the Claus furnace (Item 1) with a stream of oxygen from the air separation unit. The use of oxygen, rather than air, makes the Claus reactors smaller and lower in cost. This burner is operated at sufficiently high temperature, >1427°C (>2600°F) to promote decomposition of ammonia, and also converts most of the contained H<sub>2</sub>S to SO<sub>2</sub>. The burner off-gas is mixed with the untreated acid gas, the proportions of H<sub>2</sub>S and SO<sub>2</sub> being optimized for Claus reactor conversion. The gas is then cooled in a boiler (Item 3) to generate IP-steam, followed by a boiler-sulfur condenser (Item 4) generating LP-steam. Any elemental sulfur contained in the product acid gas is separated before the gas enters the first Claus reactor (Item 5). Three stages of acid gas reheat, with IP-steam heat source, Claus catalytic reactor, gas cooling to generate LP-steam, and sulfur separation are used in the process. The sulfur recovery process recovers about 91.60% of the sulfur content of the acid gas. The collected sulfur streams are combined and the sulfur is stored for treatment and marketing as a by-product.

The tail gas has a high content, and variety of sulfur species ( $H_2S$ ,  $SO_2$ ,  $S_x$ ), and the most effective way the power plant can achieve its overall 99% sulfur removal goal is for this tail gas to be recompressed and recycled to the gasifier. After cooling and compression and condensate removal, the tail gas recycled to the Gasifier has the composition reported in Table 4.14. The fates of the contaminants in the Sulfur Recovery Section are listed in Table 4.15.

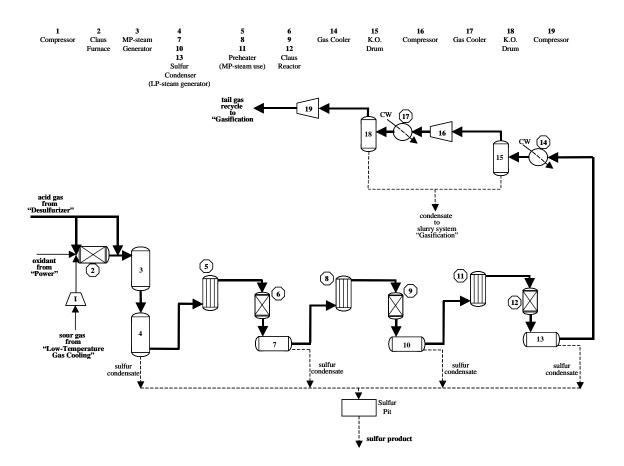


Figure 4.5 - Sulfur Recovery Section with Conventional Technology

				-	
Stream name	Acid Gas to	Oxidant from	Sour gas	Sulfur	Tail gas
	Sulfur	Power Island		product	recycle
	Recovery				
Molar flow, kmole/hr	375	42	11	132	278
Mass flow, kg/hr	15,076	1,346	259	3805	11,354
Volumetric flow, m <sup>3</sup> /hr	5,156	133	193	2.4	312
Temperature, °C	38	27	62	124	221
Pressure, kPa	186	793	103	153	3620
Enthalpy, MJ/hr	-1.04E+05	0.44	-2327	-18157	-92511
Molecular wt	40.2	31.9	23.8	28.8	40.9

 Table 4.13 – Stream Characteristics for Sulfur Recovery Section

	Major constituents (vol%)
$H_2$	2.91
СО	9.02
$CO_2$	82.38
$H_2O$	1.12
$N_2$	1.11
Ar	0.08
$H_2S$	2.09
$SO_2$	0.98
COS	0.28
NH <sub>3</sub>	0.00
HCN	0.02
Total	99.99

Table 4.14 - Recycle Tail Gas Composition – Conventional Technology with Current Standards

Table 4.15 - Fate of Contaminants in Conventional Sulfur Recovery - Current Standards

Contaminant	Removal	Contaminant conversions
	(% of inlet to Section)	
Sulfur species	91.60	Claus conversion to elemental
_		sulfur
		Hydrolysis to reduced forms
Halides	NA	
Ammonia	99.0	Furnace decomposition to N <sub>2</sub>
Hydrogen cyanide	NA	
Mercury	NA	

# **Power Section**

The Power Section process flow diagram is shown in Figure 4.6. Characteristics of major streams in the Power Section are listed in Table 4.16. An oxygen stream with 95% purity, is generated by conventional, pressurized, cryogenic air separation (ASU). The air separation unit's input air is totally supplied as pressurized air extracted from the turbine air compressor. A relatively low-purity nitrogen stream is also produced that is compressed (Item 19) and humidified to be used for clean fuel gas dilution. A smaller stream of high-purity nitrogen (99.9%) is also produced that is compressed (Item 20, Item 22) to be used for various small process needs including filter pulse cleaning. The oxygen feed rate differs slightly between the two evaluation cases due to the consumption of oxygen needed to operate the gasifier with recycled streams, and oxygen consumed by the sulfur recovery process, with the conventional, low-temperature gas cleaning technology. Low-purity nitrogen is humidified (Item 24) to about 15 vol% water vapor using low-grade heat sources (Item 16, Item 21), and the nitrogen stream is mixed with the clean fuel gas before the mixture is distributed to the gas turbine combustors.

The turbine combustors operate with an outlet temperature of about 2770°F, and, with the peak flame temperature being about 3000°F, the NOx emission is expected to be less than 15 ppmv (dry, corrected to 15% oxygen). The turbine exhaust gas has a temperature of 1152°F. The exhaust gas passes through the heat recovery steam generator (HRSG – Item 5), generating a superheated, high-pressure steam flow of 337,698 kg/hr (744,500 lb/hr). The stack gas from the power plant has a temperature of

about 121°C (250°F) and a composition listed in Table 4.17. The fates of the contaminants in the Power Section are listed in Table 4.18.

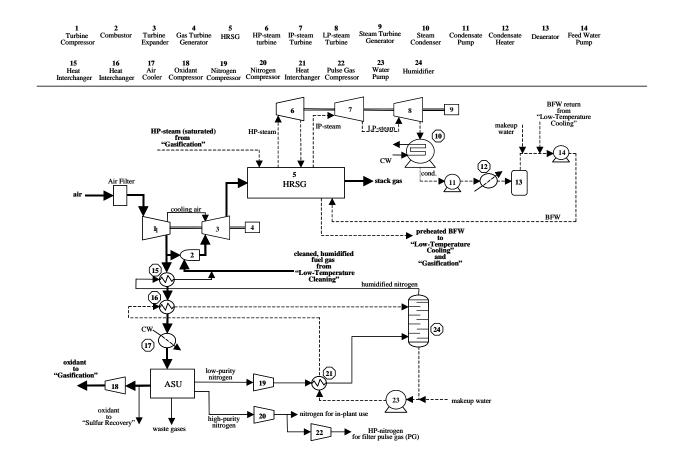


Figure 4.6 - Power Section – Conventional Technology

Stream name	Air to gas	Gasifier	Low-purity	Makeup	Fuel gas to	Stack gas
	turbine	oxidant	nitrogen	water	gas turbine	
Molar flow, kmole/hr	53,609	2,899	10,650	1,863	10,166	59,035
Mass flow, kg/hr	1,546,965	92,357	299,941	33,566	213,005	1,696,586
Volumetric flow, m <sup>3</sup> /hr	1,278,821	2,377	33,514	34	22,934	1,911,005
Temperature, °C	15	81	27	15	310	122
Pressure, kPa	100	3585	793	2068	2,158	101
Enthalpy MJ/hr	-1.52E+05	4,283	108	-5.34E+05	-1.26E+06	-3.63E+06
Molecular wt	28.9	31.9	28.2	18.0	21.0	28.7

1 abic 4.10 - Bu cam Characteristics for 1 ower beenon	Table 4.16 –	Stream	Characteristics	for	Power	Section
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	Major constituents (vol%)
O <sub>2</sub>	8.06
$CO_2$	9.19
H <sub>2</sub> O	11.68
N <sub>2</sub>	70.22
Ar	0.84
Total	99.99
	Major contaminants
SO <sub>2</sub> (ppmv)	17.7
NOx (ppmv)	15
Mercury (ppbv)	0.05

Table 4.17 - Stack Gas Composition - Current Standards

Table 4.18 – Fate of Contaminants in Conventional Power Section – Current Standards

Contaminant	Removal	Contaminant form conversion
	(% of inlet to Section)	
Sulfur species	0	Oxidation to SOx
Halides	NA	
Ammonia	0	Partial oxidation conversion to NOx
Hydrogen cyanide	NA	
Mercury	0	Partial conversion to oxidized forms

# 4.2 IGCC WITH CONVENTIONAL GAS CLEANING TO MEET FUTURE STANDARDS

IGCC using conventional, low-temperature gas cleaning technology to meet Future Standards has a very similar process flow configuration to the power plant meeting Current Standards, with only the process flow diagram for the AGR Section being different. The process stream flow rates and compositions are slightly different from the Current Standards case in most of the process sections, especially in the AGR Section. Where the process section descriptions are identical to those provided for the Current Standards case, only the modified flows and compositions are indicated.

# **Gasification Section**

Figure 4.2 shows the process flow diagram for the Gasification Section. The flows for the Future Standards case differ only slightly from those estimated for the Current Standards case. Table 4.19 lists characteristics of major streams in the Gasification Section. The raw gas flow rate at the exit of the Convective Cooler (Item 7) is about 245,350 kg/hr (540,907 lb/hr), with heat content of 540 MW(t) (1,843 x 10<sup>6</sup> Btu/hr) (LHV) and heating value of about 7.51 MJ/Nm<sup>3</sup> (191 Btu/scf), and includes 9,979 kg/hr (22,000 lb/hr) of recycled, clean fuel gas used as soot blower gas in the raw gas heat exchangers. The raw gas has the estimated composition in Table 4.20.

Stream name	Coal feed	Total slurry	Gasifier	Raw fuel	Slag	HP-steam
		water feed	oxidant	gas		
Molar flow, kmole/hr	8,810	2,429	2,902	11,437	224	15,447
Mass flow, kg/hr	98,847	43,767	92,490	247,913	10,511	278,279
Volumetric flow, m <sup>3</sup> /hr	122	45	2,352	22,709	13	5,123
Temperature, °C	26	56	84	371	670	318
Pressure, kPa	101	110	3654	2703	2537	10,583
Enthalpy MJ/hr	-1.25E+05	-6.89E+05	4,495	-1.49E+06	-41,7031	-3.69E+06
Molecular wt	11.2	18.0	31.9	21.7	46.9	18.0

 Table 4.19 – Stream Characteristics for Gasification Section - Future Standards

Table 4.20 - Raw Gas Composition - Future Standards

	Major constituents (vol%)
H <sub>2</sub>	23.65
CH <sub>4</sub>	0.31
СО	40.97
CO <sub>2</sub>	10.90
H <sub>2</sub> O	21.38
N <sub>2</sub>	1.58
Ar	0.14
Total	98.88
	Major contaminants (ppmv)
H <sub>2</sub> S	9,578
COS	726
$CS_2$	0.7
S <sub>X</sub>	8
$SO_2$	4
NH <sub>3</sub>	676
HCN	20
HCl	425
Hg (ppbv)	3

# **Low-Temperature Cooling Section**

Figure 4.3 represents the process flow diagram for the Low-Temperature Cooling Section and flows differ slightly from the Current Standards case. Table 4.21 lists characteristics of major streams in this section.

Stream name	Cooled	Slag/char	Fuel gas to	Desulfurized	Reheated fuel gas
	raw gas	recycle	Desulfurization	fuel gas	to Power Island
Molar flow, kmole/hr	11,437	43	8,536	8,162	10,159
Mass flow, kg/hr	247,913	2,562	191,746	176,981	212,968
Volumetric flow, m <sup>3</sup> /hr	22,709	2.0	9,032	9,092	23,289
Temperature, °C	371	371	38	18	310
Pressure, kPa	2,703	2,537	2,441	2,165	2,124
Enthalpy, MJ/hr	-1.49E+06	-12,177	-9.66E+05	-8.68E+05	-1.26E+06
Molecular wt	21.7	46.9	22.5	21.7	21.0

 Table 4.21 – Stream Characteristics for Low-Temperature Cooling Section – Future Standards

<u>Barrier filter</u>: A ceramic, or metal, candle barrier filter (Item 3) operates at about  $370^{\circ}$ C ( $700^{\circ}$ F) to removal particulate (solidified slag particles and char) from the raw gas to a level of < 0.1 ppmv as the first step in the cleaning process.

<u>Fuel gas coolers</u>: A process heat interchanger (Item 4) is now used to cool the gas to about 228°C (442°F), while reheating the clean fuel gas to about 310°C (590°F) before it goes to the gas turbine combustors. This cooled gas then passes through a second heat interchanger (Item 5) that cools it to about 198°C (389°F).

<u>Gas condensate scrubber</u>: The gas is next scrubbed in a column (Item 6) with collected process condensate to remove halides to a very low level, and results in the further cooling of the gas to about  $158^{\circ}C$  ( $317^{\circ}F$ ).

<u>Gas reheater</u>: The gas is now reheated in a fuel gas heat interchanger (Item 5) to about 186°C (367°F) in preparation for COS hydrolysis.

<u>COS hydrolysis</u>: The reheated gas is treated in a catalytic reactor (Item 7) to hydrolyze its COS content to  $H_2S$ , allowing more efficient sulfur removal to be performed downstream, and to prevent HCN accumulation in the AGR solvent.

<u>Gas cooling and ammonia removal</u>: Next, a series of process heat interchangers and water-cooled heat exchangers (Items 9, 12, 14, and 17) are used to cool the gas to about 38°C (100°F), simultaneously removing most of the gas water content and collecting process condensate that contains most of the fuel gas ammonia. The stream of released gases from condensate processing is sent to the Sulfur Recovery Section of the plant. Its composition is listed in Table 4.22.

	Constituents (vol%)
$H_2$	1.12
$CH_4$	0.03
CO	2.11
CO <sub>2</sub>	17.12
H <sub>2</sub> O	47.50
N <sub>2</sub>	0.06
H <sub>2</sub> S	7.90
COS	0.04
NH <sub>3</sub>	24.11
HCN	0.00
Total	99.99

Table 4.22 - Sour-water	Gas Composition -	<b>Future Standards</b>
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<u>Mercury removal</u>: The gas is treated in a fixed bed reactor (Item 24) containing sulfur-impregnated, activated carbon to remove 95% of the mercury. The composition of the gas sent to the AGR Section is listed in Table 4.23.

	Major constituents (vol%)			
$H_2$	29.99			
CH <sub>4</sub>	0.39			
СО	51.97			
$CO_2$	13.90			
H <sub>2</sub> O	0.27			
$N_2$	2.00			
Ar	0.17			
Total	98.69			
	Major contaminants (ppmv)			
$H_2S$	13,081			
COS	0.0			
NH <sub>3</sub>	0.5			
HCN	0.0			
Hg (ppbv)	0.14			

 Table 4.23 - Gas Composition to AGR Section - Future Standards

<u>Humidification & Fuel gas reheat</u>: The clean and dry-gas from the desulfurizer is reheated to about 141°C (286°F), interchanging heat (Item 12) with the previous gas cooling-condensation process streams. The cleaned and humidified fuel gas is now reheated to about 310°C (590°F) in a heat interchanger (Item 4).

### Acid Gas Recovery (AGR) Section

Figure 4.7 shows the process flow diagram for the AGR Section with the Future Standards. Table 4.24 lists characteristics of major streams in the AGR Section. The Rectisol desulfurization technology is selected for the desulfurization step, with other commercial low-temperature desulfurization technologies, such as Selexol, unable to meet the stringent requirements (Sharp et al., 2003). Rectisol claims that this process will also effectively remove HCN, NH<sub>3</sub>, CS<sub>2</sub>, iron and nickel carbonyls, VOCs, and mercury effectively (Koss and Meyer, 2003). On the other hand, users of Rectisol perform mercury removal in a separate unit placed before the Rectisol process, and place adsorbers to capture metal carbonyls after the Rectisol process to ensure performance and because if these contaminants are absorber in the Rectisol solvent, will either accumulate within the solvent or be released from the stripper and need separate processing to engage their final disposition. Maintenance of the Rectisol columns is a critical path item in an IGCC power plant using this technology due to the severe conditions in the columns (Trapp et al., 2004).

The cooled gas, following mercury removal, is desulfurized in a conventional, Rectisol-based absorption-stripping process. The Rectisol process contacts gas recuperatively-cooled to about -27°C (-17°F) (Item 1) with refrigerated methanol at about -63°C (-82°F) (Item 8) in a multi-staged, bubble-cap tray, counter-current absorption column (Item 3). A small portion of methanol is injected directly into the gas stream before the heat interchanger Item 1 to remove water from the gas (Item 2) to eliminate the possibility of ice formation. The desulfurized gas meets the requirement for an overall 99.98% sulfur removal performance in the power plant, if there are no other sulfur release points in the power plant. The desulfurized, reheated gas exits the process (Item 1) at about 18°C (64°F). Makeup methanol solvent (141 lb/hr) is fed to the process to account for process losses, and a methanol decontamination step is included in the process. The process uses significant energy for refrigeration associated with heat

interchanger Item 8 and condenser Item 12 to meet this level of sulfur removal. The lean solvent is flashed in a series of flash tanks (Item 6 and 7) to low pressure (15 psia) and is circulated to a solvent stripper column (Item 11). The stripper generates a low-pressure, acid gas that is sent to a sulfur recovery process. Table 4.25 lists the composition of the desulfurized fuel gas.

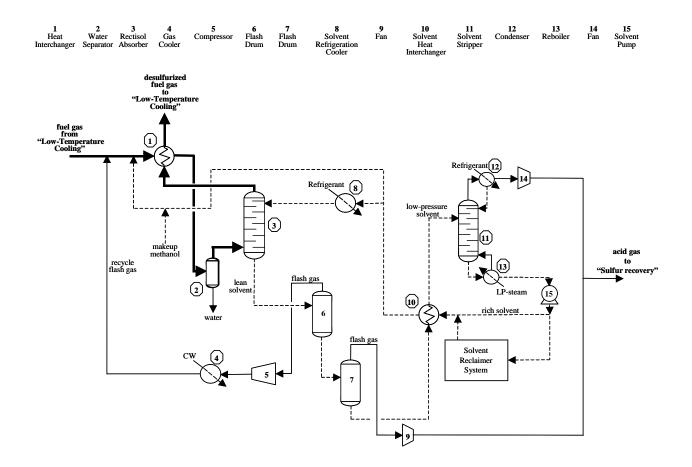


Figure 4.7 – AGR Section – Rectisol for Future Standards

Stream name	Fuel gas from	Desulfurized	Acid Gas to
	LT-Cooling	fuel gas	Sulfur Recovery
Molar flow, kmole/hr	8,536	8,162	353
Mass flow, kg/hr	191,746	176,986	14,406
Volumetric flow, m <sup>3</sup> /hr	9,032	9,093	4,902
Temperature, °C	38	18	41
Pressure, kPa	2,441	2,165	186
Enthalpy, MJ/hr	-9.66E+05	-8.75E+05	-0.97E+05
Molecular wt	22.5	21.7	40.8

Table 4.24 – Stream Characteristics for AGR Section – Future Standards

	Major constituents (vol%)			
H <sub>2</sub>	31.37			
CH <sub>4</sub>	0.41			
СО	54.35			
CO <sub>2</sub>	11.60			
H <sub>2</sub> O	0.00			
N <sub>2</sub>	2.09			
Ar	0.18			
Total	100.00			
	Major contaminants (ppmv)			
$H_2S$	2.1			
COS	0.5			
NH <sub>3</sub>	0.0			
HCN	0.0			
Hg (ppbv)	0.12			

 Table 4.25 - Desulfurized Fuel Gas Composition - Future Standards

Table 4.26 lists the composition of the acid gas sent to the Sulfur Recovery Section. Note that some methanol solvent is estimated to be lost to the acid gas stream.

	Major constituents (vol%)
$H_2$	0.00
CO	0.02
CO <sub>2</sub>	67.93
H <sub>2</sub> O	0.00
$H_2S$	31.51
COS	0.00
NH <sub>3</sub>	0.00
HCN	0.00
methanol	0.53
Total	99.99

# Table 4.26 - Acid Gas Composition - Future Standards

Table 4.27 summarizes the fates of the contaminants within the AGR process for the Future Standards case. The fuel gas composition after humidification is listed in Table 4.28.

Contaminant	Removal	Contaminant conversions
	(% of inlet to Section)	
Sulfur species	99.981	Methanol absorption
Halides	NA	
Ammonia	99.99	None
Hydrogen cyanide	NA	
Mercury	100	Accumulates in methanol

	Major constituents (vol%)			
H <sub>2</sub>	25.20			
CH <sub>4</sub>	0.33			
CO	43.66			
CO <sub>2</sub>	9.32			
H <sub>2</sub> O	19.66			
N <sub>2</sub>	1.68			
Ar	0.14			
Total	99.99			
	Major contaminants (ppmv)			
$H_2S$	1.7			
COS	0.4			
NH <sub>3</sub>	0.0			
HCN	0.0			
Hg (ppbv)	0.146			

Table 4.28 - Cleaned and Humidified Fuel Gas Composition - Future Standards

#### **Sulfur Recovery Section**

Figure 4.5 shows the process flow diagram for the Sulfur Recovery Section of the IGCC power plant. The stream flows and conditions are very similar to those for the Current Standards case. The sour-water gas, containing significant ammonia and  $H_2S$ , and an appropriate portion of the acid gas are combined and are burned in the Claus furnace (Item 1) with a stream of oxygen at 1,352 kg/hr (2,980 lb/hr) from the air separation unit. The sulfur recovery process recovers about 92.6% of the sulfur content of the acid gas. The collected sulfur streams are combined and the sulfur is stored for treatment and marketing as a by-product.

The tail gas, at 10,800 kg/hr (23,811 lb/hr), contains a considerable content, and variety of sulfur species ( $H_2S$ ,  $SO_2$ ,  $S_x$ ), and the only way the power plant can achieve its overall 99.85% sulfur removal goal is for this tail gas to be recompressed and recycled to the gasifier. After cooling and compression and condensate removal, the tail gas recycled to the Gasifier has the composition reported in Table 4.29.

	Major constituents (vol%)
$H_2$	3.15
СО	10.36
$CO_2$	80.37
H <sub>2</sub> O	1.00
$N_2$	1.16
Ar	0.08
$H_2S$	2.21
$SO_2$	1.04
COS	0.39
NH <sub>3</sub>	0.00
HCN	0.03
methanol	0.20
Total	99.99

 Table 4.29 - Recycle Tail Gas Composition - Future Standards

### **Power Section**

The Power Section process flow diagram is shown in Figure 4.6. The characteristics of major streams are listed in Table 4.30. An oxygen stream with 95% purity, is generated by conventional, pressurized, cryogenic air separation (ASU). Low-purity nitrogen is humidified to about 15 vol% water vapor using low-grade heat sources, and the nitrogen stream is mixed with the clean fuel gas before the mixture is distributed to the gas turbine combustors.

Stream name	Air to gas	Gasifier	Low-purity	Makeup	Fuel gas to	Stack gas
	turbine	oxidant	nitrogen	water	gas turbine	
Molar flow, kmole/hr	53,550	2,903	10,666	1,863	10,159	58,967
Mass flow, kg/hr	1,545,243	92,490	300,385	33,566	212,968	1,694,683
Volumetric flow, m <sup>3</sup> /hr	1,277,397	2,352	33,563	34	23,289	1,905,963
Temperature, °C	15	84	27	15	310	121
Pressure, kPa	100	3654	793	2068	2,124	101
Enthalpy MJ/hr	-1.52E+05	4,495	108	-5.34E+05	-1.26E+06	-3.63E+06
Molecular wt	28.9	31.9	28.2	18.0	21.0	28.7

Table 4.30 – Stream Characteristics for Power Section – Future Standards

The turbine combustors, advanced, low-NOx burners specifically designed for low heating-value fuel gas, operate with an outlet temperature of about 1529°C (2784°F), and with the peak flame temperature being less than 1649°C (3000°F), the NOx emission is expected to be less than 5 ppmv (dry, corrected to 15% oxygen). The turbine exhaust gas has a temperature of 622°C (1152°F). The exhaust gas passes through the heat recovery steam generator (HRSG), generating a superheated, high-pressure steam flow of 346,996 kg/hr (765,000 lb/hr). The stack gas from the power plant has a temperature of 121°C (250°F) and a composition listed in Table 4.31. While the major constituents in the stack gas are very similar to those in the Current Standards case (Table 4.17), the major contaminants are significantly lower in the Future Standards case.

	Major constituents (vol%)
<b>O</b> <sub>2</sub>	8.04
CO <sub>2</sub>	9.20
H <sub>2</sub> O	11.68
$N_2$	70.23
Ar	0.84
Total	99.99
	Major contaminants
$SO_2(ppmv)$	0.4
NOx (ppmv)	5
Mercury (ppbv)	0.026

Table 4.31 - Stack Gas Composition - Future Standards

### 4.3 CONVENTIONAL IGCC POWER PLANT PERFORMANCE ESTIMATES

The breakdown of power generation and power use in the IGCC power plant with conventional, low-temperature, dry-gas cleaning technology is shown in Table 4.32. For the two gas cleaning cases, all

of the sections of the power plant have similar total power use, except for the AGR Section where the refrigeration power needed in the Future Standards case is significantly greater than the power use for the AGR Section in the Current Standards case. Overall, the lower sulfur emission of the Future Standards case results in an increase in plant heat rate of about 3.5%, a reduction in the power plant thermal efficiency of about 1.3 percentage-points, and a reduction in net plant generating capacity of about 9.7 MWe.

Section	Current Standards	Future Standards	
	Power (MWe)	Power (MWe)	
Power Island Generation			
Turbine air compressor	-169.8	-169.5	
Gas turbine	365.8	365.2	
Steam turbine	137.9	139.6	
Generator	-6.7	-6.7	
BOP	-4.8	-4.9	
Total power	322.4	323.7	
Air Separation Consumption			
ASU Air compressor	0	0	
Oxygen compressor	4.19	4.26	
Nitrogen compressor	9.04	9.05	
ASU	20.60	20.64	
Total power use	33.83	33.95	
Gasification Consumption			
Fans & blowers	0.6	0.5	
Pumps	0.10	0.10	
Coal handling and preparation	1.5	1.5	
Ash handling	1.3	1.3	
Total power use	3.5	3.4	
Desulfurization Consumption			
Refrigeration	0.0	10.77	
Compressors	0.004	0.25	
Pumps	0.19	0.12	
Total power use	0.2	11.14	
Sulfur Recovery Consumption			
Compressors	0.97	0.95	
Pumps	0.00	0.00	
Total power use	0.97	0.95	
Total Plant			
Net plant power generated, MW	285.2	275.5	
Plant net heat rate (HHV), kJ/kWh (Btu/kWh)	9579 (9079)	9915 (9397)	
Plant net efficiency, (%, LHV)	40.56	39.19	
Plant net efficiency, (%, HHV)	37.58	36.31	

Table 4.33 lists several quantities related to the use of resources and emissions in the IGCC power plant with conventional, low-temperature, dry-gas cleaning technology. The power plants in both cases use large quantities of LP-steam in the gas cleaning process steps. Cooling water use in the processing steps is also very large. Large quantities of process condensate are generated in both cases,

primarily in the Low-Temperature Cooling Section. A moderate level of fresh process water is required in the gas cleaning processes, and both cases result in a significant discharge of excess process water. Both cases require large quantities of boiler-quality makeup water for fuel gas humidification.

	Current Standards	Future Standards
Auxiliaries		
Net IP steam use, MJ/hr (10 <sup>6</sup> Btu/hr)	33,025 (31.3)	32,603 (30.9)
Net LP steam use, MJ/hr (10 <sup>6</sup> Btu/hr)	64,783 (61.4)	51,383 (48.7)
Total cooling water use, MJ/hr (10 <sup>6</sup> Btu/hr)	251,641 (238.5)	199,625 (189.2)
Net condensate generated, kg/hr (lb/hr)	1,523 (3,357)	1849 (4,077)
Total process water used, kg/hr (lb/hr)	988 (2,178)	680 (1,499)
Net process water discharge, kg/hr (lb/hr)	274 (603)	0
Total boiler-water makeup, kg/hr (lb/hr)	69,263 (152,696)	69,646 (153,540)
Emissions		
Sulfur total removal efficiency (%)	99.08	99.98
Sulfur total emission, mg/MJ (lb $SO_2/10^6$ Btu)	24.4 (0.0568)	0.52 (0.0012)
Sulfur total emission, kg/MW (lb SO <sub>2</sub> /MW)	0.23 (0.516)	0.005 (0.011)
NOx total emission, mg/MJ (lb $NO_2/10^6$ Btu)	14.9 (0.0347)	4.7 (0.0110)
Particulate emission, mg/MJ (lb/10 <sup>6</sup> Btu)	0.0073 (1.708E-05)	0.0073 (1.701E-05)
Hg emission, mg/MJ (lb/TBtu)	0.22 (0.51)	0.11 (0.26)
Consumibles, kg/hr (lb/hr)		
Chemicals	34 (75)	64 (142)
Sorbents	1.4 (3)	1.4 (3)
Catalysts	0.9 (2)	0.9 (2)
Solid waste, kg/hr (lb/hr)		
Slag product (25 wt% water)	14,015 (30,897)	14,015 (30,897)
Waste salts (25 wt% water)	285 (628)	285 (628)
Sorbent wastes (hazardous)	1.4 (3)	1.4 (3)
Total solid waste	14,301 (31,528)	14,301 (31,528)

Table 4.33 - IGCC Power Plant Resource Use and Emissions with Conventional Gas Cleaning

The total sulfur emissions are expressed under three different bases: percent removal, mg/MJ (lb  $per10^{6}$  Btu) of fuel input, and kg/MWe ( lb per MWe) of net power generated. The sulfur emissions are significantly lower for the Future Standard case, and are comparable to the sulfur emissions from a natural gas-fired, combined-cycle power plant. Solid waste rates from the IGCC power plants are identical in both cases.

# 4.4 CONVENTIONAL IGCC PLANT COST ESTIMATES

The major equipment purchase costs and installed costs associated and sensitive to the gas cleaning process are listed in Table 4.34 for each of the sections of the power plant evaluated. The cost of the Convective Cooler from the Gasification Section, and the Mercury Removal cost has been taken out and reported as separate items. The Low-temperature Cooling Section is the most expensive of the gas cleaning sections. Its cost is approached by the cost of the AGR Section for the Future Standards case. The total gas cleaning costs are also reported on the basis of dollars per kilowatt of net power generated. The equipment costs are about 50% greater for the Future Standards case than for the Current Standards case, reflecting primarily the great cost increase associated with increased sulfur removal performance of the gas cleaning system. The technical difficulty of achieving the sulfur removal standards are reflected

in the percent of the feed sulfur emitted to the stack in each plant: 1% of the raw gas sulfur is emitted in the Current Standards case versus a 0.02% sulfur emission in the Future Standards case.

Plant Section	Current Standards	Future Standards		
	Cost, k\$	Cost, k\$		
Raw Gas Convective Cooling				
purchased equipment	902	960		
installed equipment	1,802	1,919		
Low-temperature Gas Cooling				
purchased equipment	6,550	6,421		
installed equipment	11,679	11,623		
AGR				
purchased equipment	1,143	7,663		
installed equipment	2,459	12,860		
Sulfur Recovery				
purchased equipment	2,531	2,839		
installed equipment	4,711	5,228		
Mercury Removal				
purchased equipment	1,018	1,036		
installed equipment	1,482	1,540		
Total Gas Cleaning				
purchased equipment	12,143	18,919		
installed equipment	22,132	33,169		
purchased equipment, \$/kW	43	69		
installed equipment, \$/kW	78	120		
Total Plant				
TCR, k\$	427,748	444,703		
TCR, \$/kW	1,500	1,614		

 Table 4.34 - Conventional Gas Cleaning Technology Equipment Cost Breakdown

The Total Capital Requirements for the IGCC power plants using conventional, low-temperature, dry-gas cleaning technology are also computed in Table 4.34. The costs are, in part, based on the assumption that an IGCC power plant of this capacity and scope, using conventional gas cleaning technology to meet current standards, has a representative Total Capital Requirement of 1,500 \$/kW (Holt, 2003). For the Standard gas cleaning requirements, the conventional gas cleaning process TCR is about 7.8% of the total plant TCR. For Future gas cleaning standards, the conventional gas cleaning process TCR is about 11.2% of the total plant TCR. On a total power plant basis, the Total Capital Requirement (\$/kW) for the Future Standards case is about 8.0% greater than for the Current Standards case.

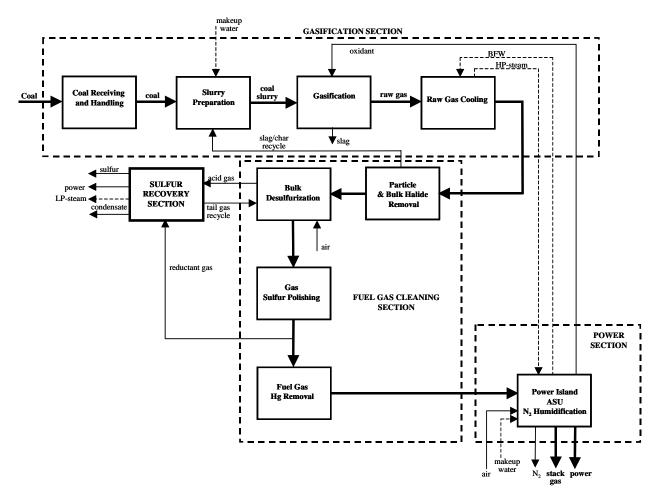
The cost-of-electricity (COE) for the IGCC power plant for the Current Standards and Future Standards cases are also computed in Table 4.35. The COE of the power plant under the Future Standards case is 5.6% greater than that for the Current Standards case. The capital charges are the dominant cost factor, with the fuel cost and O&M cost being comparable.

	Current Standards	Future Standards
Fixed O&M	0.51	0.53
Variable O&M	0.27	0.27
Consumables	0.21	0.21
water	0.04	0.04
chemicals, sorbents, catalysts	0.032	0.027
waste Disposal	0.108	0.112
chemicals for BFW, CW and	0.03	0.03
waste treatment		
Sulfur by-product credit	0.063	0.066
Fuel	1.68	1.74
Capital charges	3.95	4.25
Total	6.56	6.93

 Table 4.35 – Cost-of-Electricity – Conventional Gas Cleaning

### 5. IGCC WITH FILTER-REACTOR NOVEL GAS CLEANING

The IGCC power plant process configuration using the Filter-Reactor Novel Gas Cleaning Process differs considerably from the conventional IGCC power plant, and these differences result in significantly improved power plant performance and cost. A simplified process flow diagram for IGCC with Novel Gas Cleaning is shown in Figure 5.1. It portrays the relations between the major process systems in the plant and shows only the major process streams.



#### Figure 5.1 – Overall IGCC Power Plant Scheme with Filter-Reactor Novel Gas Cleaning

The overall process schematic is broken into major plant sections: the Gasification Section, consisting of coal receiving and handling, slurry preparation, gasification, slag handling, and raw gas cooling sub-sections; the Desulfurization Section, which includes mercury removal, the Sulfur Recovery Section, and the Power Section, consisting of the Power Island and air separation sub-sections. The diagram is highly simplified, not showing all of the numerous sub-functions of each section and the numerous streams that pass between the various sections.

Unlike the conventional, low-temperature, dry-gas cleaning diagram, in Figure 4.1, Figure 5.1 has no Low-temperature Cooling Section and the gas is maintained above its dew-point temperature – this is designated "humid-gas" cleaning. Thus, there is no large recycle water stream sent to the slurry preparation section, no sour-water gas stream sent to Sulfur Recovery, and no halide-salt disposal product appears. Also, fuel gas humidification is not needed. Sulfur Recovery tail gas is recycled to the

Desulfurization Section rather than to the Gasification Section as it is with conventional gas cleaning, and a small stream of clean gas is sent to the Sulfur Recovery Section. Mercury Removal follows directly after desulfurization, and sorbents are fed to the Desulfurization and Mercury Removal sections, with associated sorbent wastes withdrawn from each of these sections.

Detailed material & energy balances have been developed for the several sections that relate directly to gas cleaning and its impacts on the power plant performance: Gasification, Desulfurization, Sulfur Recovery, and the Power Section.

The major functions of each gas cleaning-related sections are:

Novel Gas Cleaning Gasification Section (Figure 5.2):

- prepare coal slurry
- accept recycled slag
- gasify coal slurry
- cool raw gas, generating saturated, HP-steam
- cool and separate slag from water for disposal

Fuel Gas Cleaning Section (Figure 5.3):

- remove raw gas particulate (slag) and recycle to Gasification Section
- remove raw gas halide
- accept tail gas recycle from Sulfur Recovery Section
- desulfurize gas
- produce suitable acid gas
- reheat cleaned fuel gas
- remove mercury (once-through or regenerative sorbent systems)
- split off clean gas stream for compression as soot blower gas
- split off clean gas stream for Sulfur Recovery Section reductant

Sulfur Recovery Section (Figure 5.4):

- acid gas expansion and power recovery
- acid gas partial reduction to desired H<sub>2</sub>S-SO<sub>2</sub> gas mixture
- elemental sulfur production
- tail gas recycle to Desulfurization Section
- condensate recycle to Gasification Section
- IP-steam and LP-steam production

Power Section (Figure 5.5):

- air compression for air separation and fuel gas combustion
- air separation to generate gasifier oxidant, and nitrogen streams
- low-purity nitrogen stream humidification and mixing with clean fuel gas
- fuel gas combustion for low-NOx production and expansion for power generation
- gas turbine exhaust gas heat recovery steam generation
- steam expansion for power generation
- circulate BFW to Gasification Section and Desulfurization Section

# 5.1 FATE OF CONTAMINANTS WITH NOVEL GAS CLEANING TECHNOLOGY

Coal contains significant content of a large number of trace elements, including all of those listing in Tables 3.2 and 3.3, with their associated fuel gas cleaning requirements. To estimate what content these trace elements might have in the gas, and in what forms and phases (vapor, liquid, or solid), the NASA-Lewis Chemical Equilibrium Program was utilized (McBride and Gordon, 1996). Average

values of Eastern-US coal trace element content were input, along with the relative input of water and oxidant, and equilibrium gas compositions were generated over the range of gas cleaning temperatures representative of the conditions seen in both conventional gas cleaning and Novel Gas Cleaning technologies. With the conventional, low-temperature, dry-gas cleaning technology, it is well known that many of the gas-phase contaminants are condensed out of the raw gas and either accumulate on process equipment, such as heat exchangers, or are trapped in process condensate streams, resulting in condensate treatment requirements and water discharge issues. In this assessment, the key trace element contents in the gas were followed from the raw gas hot outlet of the gasifier, through the various cleaning stage temperatures, all the way to the turbine combustor inlet.

Table 5.1 lists the results, showing what compound-forms of each elements might be present, where condensed forms of these elements might be removed in the process, what forms might reach the gas turbine in the clean fuel gas, and what content they might have at the conditions of the lowest-temperature section, the Mercury Removal Section, and on to the turbine combustors. Only Hg, and Ni and Fe in carbonyl forms are of concern. It is expected that 90-95% mercury removal, in the form of elemental mercury, will be required by the mercury removal section. It is possible that some nickel removal, in the form of nickel carbonyl, might also result in the mercury removal stage.

It is estimated that Ni-carbonyls will not form above about 480°C (900°F), and Fe-carbonyls above about 220°C (400°F), and conversely, once formed they will decompose when raised above these temperatures. Potential Ni and Fe sources are the coal ash constituents and the process equipment and piping. In the Novel Gas Cleaning process, in contract to conventional gas cleaning technology, the coal ash constituents are removed from the gas at temperatures above those where metal carbonyls can form, so this source is eliminated. Also in the Novel Gas Cleaning process, some Ni-carbonyls could form in the lowest temperature cleaning stages on contact with Ni-containing equipment and piping, but these Nicarbonyls will decompose in subsequent reheat steps, protecting the turbine from damage. The amount of nickel carbonyl that will form in the actual process is uncertain, but it is likely that much less will form at the Novel Gas Cleaning conditions than will form at the conventional gas cleaning conditions. It is also very likely that no iron carbonyls will form when using the Novel Gas Cleaning process.

The other trace element forms listed in Tables 3.2 and 3.3 are condensed out in the gasifier slag, in the halide filter-reactor, in the bulk sulfur removal system, in the polishing sulfur removal filter-rector or in the mercury removal system. As and Se are the only other forms prevalent in the clean fuel gas and are not currently regulated. Further, specific removal steps are not needed.

As part of the process requirements for the evaluation, the final disposition of the contaminants captured and the sorbents fed in the Novel Gas Cleaning Process are also specified. The captured contaminant's final dispositions are:

- sulfur: converted to elemental sulfur by-product,
- halides: tied up as dry solid salts with halide sorbent as non-hazardous disposal product,
- fine char/slag particulate: recycled to gasifier and incorporated into gasifier slag,
- ammonia: primarily destroyed in the staged, turbine combustors, with some conversion to NOx,
- mercury: contained in once-through sorbent,
- nickel carbonyl: if formed, is likely to plate out on clean fuel gas reheat exchangers.

The gas cleaning sorbents are fed as relatively small streams to the system and their final dispositions are:

- halide removal sorbent: once-through sorbent disposal as non-hazardous waste,
- bulk sulfur removal sorbent: regenerative sorbent, with waste fed to gasifier for slagging, or disposed as non-hazardous waste,
- polishing sulfur removal sorbent: once-through or regenerative sorbent, with waste fed to gasifier for slagging, or disposed as non-hazardous waste,
- mercury removal adsorbent: once-through sorbent disposed as hazardous waste.

	Major forms existing	Removal of	Forms in clean fuel gas	Equilibrium in
	at 220-260°C (400-500°F)	condensed forms	to turbine	gas at 220-260°C
	at 220-200 C (400-300 F)	condensed forms	to turbine	(400-500°F
				(400-300 F (ppbw)
Åa	As <sub>4</sub> , AsCl, AsN, AsH <sub>3</sub>	None	As <sub>4</sub> , AsCl, AsN, AsH <sub>3</sub>	20
As Hg	Hg	None	Hg	5
-	8		0	-
Se	Se <sub>2</sub> , SeO, SeH, SeH <sub>2</sub> , PbSe	None (possible NiSe <sub>1.05</sub> (S) in bulk sulfur removal system)	Se <sub>2</sub> , SeO, SeH, SeH <sub>2</sub>	900
Ba	BaCl <sub>2</sub> , BaF <sub>2</sub> , Ba(OH) <sub>2</sub> , BaS(CR)	BaCl <sub>2</sub> , BaF <sub>2</sub> , BaO, BaS in gasifier slag, char/halide filter, and bulk sulfur removal	Many possible	< 10 <sup>-12</sup>
Ca	CaCl <sub>2</sub> , CaF <sub>2</sub>	CaCl <sub>2</sub> in char/halide filter, bulk sulfur removal, polishing sulfur removal	CaCl <sub>2</sub> , CaF <sub>2</sub>	< 10 <sup>-12</sup>
Fe	FeS, Fe, FeCl <sub>2</sub> , Fe(OH) <sub>2</sub> , Fe(CO) <sub>5</sub> , FeS(L)	With gasifier slag and slag/halide filter	Fe(CO) <sub>5</sub>	10
K	KBr, K <sub>2</sub> Br <sub>2</sub> , K <sub>2</sub> CO <sub>3</sub> , KS, K <sub>2</sub> S, K, KCN, KCl, KF, K <sub>2</sub> Cl <sub>2</sub> , K <sub>2</sub> F <sub>2</sub> , KH, KOH, NaK	KCl and KBr liquids and solids in bulk sulfur removal and polishing sulfur removal	Many possible	< 10 <sup>-12</sup>
Li	LiCl, LiF, LiBr, Li, LiH, LiOH	LiCl, LiF in char/halide filter, bulk sulfur removal, polishing sulfur removal	Many possible	< 10 <sup>-12</sup>
Mn	MnBr, MnCl, MnS, Mn	MnS, MnC <sub>2</sub> in gasifier slag and char/halide removal filter	Many possible	< 10 <sup>-12</sup>
Na	Na <sub>2</sub> Br <sub>2</sub> , NaBr, NaK, NaS, NaCN, NaCL, NaF, Na <sub>2</sub> , Na <sub>2</sub> Cl <sub>2</sub> , Na <sub>2</sub> F <sub>2</sub> , NaS, NaSH, NaH, NaOH	NaOH, NaCl, NaF, Na <sub>2</sub> CO <sub>3</sub> , NaCN in char/halide filter, bulk sulfur removal, polishing sulfur removal	Many possible	< 10 <sup>-12</sup>
Ni	Ni(CO) <sub>4</sub> , Ni, NiS, Ni <sub>3</sub> S <sub>2</sub> (L), NiH, Ni(OH) <sub>2</sub> , NiCl,	Ni <sub>3</sub> S <sub>2</sub> in gasifier slag, char/halide filter, bulk sulfur removal	Ni(CO) <sub>4</sub>	1000
Р	HPO, P <sub>2</sub> , PH <sub>3</sub> , PO, PS, P <sub>2</sub> O <sub>3</sub> , Ca <sub>2</sub> P <sub>2</sub> O <sub>7</sub> (L), PSCl <sub>3</sub> , PCl <sub>3</sub> , PFCl <sub>2</sub> , PF <sub>3</sub>	H <sub>3</sub> PO <sub>4</sub> , Ca <sub>2</sub> P <sub>2</sub> O <sub>7</sub> in gasifier slag, char/halide filter, bulk sulfur removal	Many possible	<10 <sup>-12</sup>
Pb	PbSe, Pb, PbCl, PbS, PbH, PbBr, PbO	PbS in char/halide filter, bulk sulfur removal	PbS, PbCl, PbBr	<10 <sup>-12</sup>
V	VCl <sub>2</sub> , VOCl <sub>3</sub> , VCl <sub>4</sub> , VO <sub>2</sub>	V <sub>2</sub> O <sub>3</sub> in slag removal, char/halide filter, bulk sulfur removal	Many possible	<10 <sup>-12</sup>
Zn	ZnO, ZnS, Zn, ZnCl, ZnCl <sub>2</sub> , ZnH	ZnS in char/halide filter, bulk sulfur removal	Zn, ZnCl <sub>2</sub>	<10 <sup>-12</sup>

# Table 5.1 - Trace Component Fates in IGCC with Novel Gas Cleaning

# 5.2 IGCC WITH NOVEL GAS CLEANING TO MEET CURRENT STANDARDS

The IGCC power plant with the Filter-Reactor Novel Gas Cleaning technology must meet:

- SOx: 99% coal sulfur removal "net" for entire power plant,
- NOx: 15 ppmv (corrected to 15% oxygen, dry) at the plant stack,
- Particulate: 0.1 ppmw in the fuel gas,
- Mercury: 90% coal mercury removal,
- Power island contaminants:
  - halides: 5 ppmv in the fuel gas (after humidification, before nitrogen dilution)

- volatile metals (Ni, Fe, ): formation of metal carbonyls are unlikely in the process
- particulate: 0.1 ppmw in the fuel gas.

This control is projected to be achieved in the Novel Gas Cleaning process steps described.

### **Gasification Section**

Figure 5.2 shows the process flow diagram constructed for the Gasification Section using the Novel Gas Cleaning Process, and includes designation of the Coal Receiving and Handling System and the Slurry Preparation System. Table 5.2 lists characteristics of major streams in this section. The process diagram is very similar to the Gasification Section process flow diagram for the conventional, low-temperature gas cleaning situation, differing primarily in the fact that no tail gas is recycled to the gasifier, and little process condensate is available (only a small amount is generated during the soot blower gas cooling and within the Sulfur Recovery Section). Thus, the slurry preparation system uses mostly fresh makeup water. Oxidant from the Power Section's Air Separation Unit combines with the coal slurry to generate high-temperature, raw gas and slag streams. The raw gas flow at the exit of the Convective Cooler (Item 7) has a heat content of 539 MW(t) (1,839 x 10<sup>6</sup> Btu/hr) (LHV) and heating value of about 7.82 MJ/Nm<sup>3</sup> (199 Btu/scf), and includes about 9,979 kg/hr (22,000 lb/hr) of recycled, clean gas used as soot blower gas for the raw gas heat exchangers.



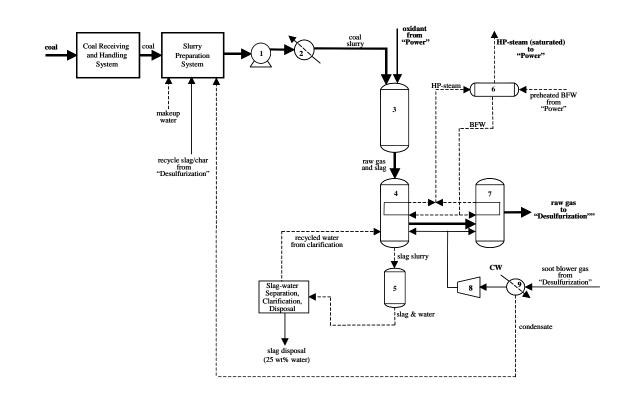


Figure 5.2 - Novel Gas Cleaning Gasification Section

The raw gas is cooled in a radiant cooler, raising saturated, high-pressure (HP) steam, and cooling the gas to about 1500°F to solidify slag particles before cooling the gas further in a convective cooler to generate additional saturated HP-steam.

Stream name	Coal feed	Total slurry	Gasifier	Raw fuel	Slag	HP-steam
		water feed	oxidant	gas		
Molar flow, kmole/hr	8,810	2,429	2,857	11,068	181	14,050
Mass flow, kg/hr	98,847	43,760	91,026	231,900	7,949	253,105
Volumetric flow, m <sup>3</sup> /hr	122	45	2,343	31,459	10	4,652
Temperature, °C	26	29	81	593	49	318
Pressure, kPa	101	110	3585	2551	110.	10,583
Enthalpy MJ/hr	-1.25E+05	-6.94E+05	4,221	-1.27E+06	-36,210	-3.35E+06
Molecular wt	11.2	18.0	31.9	21.0	43.8	18.0

Table 5.2 – Stream Characteristics for Gasification Section - Current Standards

The raw gas exits the gasifier at about 1407°C (2565°F) and 2758 kPa (400 psia), and at 593°C (1100°F) from the Convective Cooler, with the estimated composition listed in Table 5.3, not including entrained slag.

	Major constituents (vol%)
H <sub>2</sub>	25.46
CH <sub>4</sub>	0.30
CO	40.77
$CO_2$	9.73
H <sub>2</sub> O	20.59
N <sub>2</sub>	1.93
Ar	0.14
Total	98.92
	Major contaminants (ppmv)
$H_2S$	8,611
COS	634
$CS_2$	0.6
S <sub>X</sub>	7
$SO_2$	3
NH <sub>3</sub>	695
HCN	20
HC1	434
Hg (ppbv)	3

 Table 5.3 - Raw Gas Composition – Novel Gas Cleaning with Current Standards

### **Fuel Gas Cleaning Section**

Figure 5.3 shows the process flow diagram for the Fuel Gas Cleaning Section. Table 5.4 lists characteristics of major streams in the Fuel Gas Cleaning Section. The raw gas from the Gasification Section is first pre-cleaned of entrained slag particulate using a conventional cyclone (Item 1). The sequence of processing steps is described below.

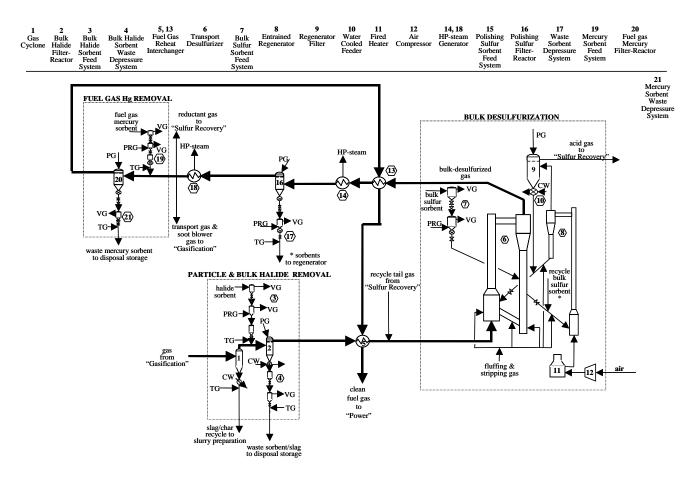


Figure 5.3 – Fuel Gas Cleaning Section - Current Standards

Stream name	Gas to	HCl	HCl	Desulfurized	Regenerator	Air to	Hg	Reheated
	cleanup	sorbent	sorbent	gas	acid gas	regenerator	sorbent	fuel gas
		feed	waste				feed	
Molar flow,	11,069	26	24	12,311	749	770	0.1	11,490
kmole/hr								
Mass flow, kg/hr	231,900	2,019	1,730	267,483	24,840	22,210	6.8	249,664
Volumetric flow,	31,460	0.9	1.8	28,972	2,752	18,360	0	34,189
m <sup>3</sup> /hr								
Temperature, °C	593	27	79	372	742	15	27	507
Pressure, kPa	2551	2758	2424	2290	2317	100	2482	2193
Enthalpy, MJ/hr	-1.27E+06	-20326	-13404	-1.44E+06	-15610	-2184.1	-34.627	-1.30E+06
Molecular wt	21	77.8	72.5	21.7	33.1	28.9	60.1	21.7

Table 5.4 – Stream Characteristics for Fuel Gas Cleaning Section – Current Standa	5.4 – Stream Characteristics for Fuel Gas Cleaning Section – (	<b>Current Standard</b>	S
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### Halide filter-reactor (Item 2):

A ceramic barrier filter is operated at about 593°C (1100°F) to remove the remaining solidified slag and char from the raw gas. Halide sorbent, -325 mesh particles of a cheap, once-through, sodium-based mineral, is fed volumetrically from a lock-hopper system (Item 3) and is mixed into the raw gas and collected in the filter-reactor. The sodium mineral reacts with the raw gas halide species, primarily HCl,

while the particles are in-flight and while they reside within the filter cake. It is estimated that more than 99% of the halides will be removed, resulting in a halide (mostly HCl) content in the fuel gas less than 5 ppmv. The halide sorbent used in the evaluation is nacholite (80 wt% sodium bicarbonate, 20 wt% inert solids), and the molar feed ratio of sodium/HCl is assumed to be 4.0 times stoichiometric. The captured slag-char, and sorbent product is drained from the halide filter-reactor, is cooled to 79°C (175°F) in a water-cooled screw conveyor (Item 4), and is depressurized and stored for disposal as a non-hazardous material. This filter-reactor provides one additional function, with the alloy-metal internals, and possibly, additional alloy surfaces installed in the vessel head, being catalytically-active for ammonia decomposition.

It is estimated that at the selected operating temperature, about 75% of the ammonia will be decomposed to nitrogen and hydrogen. A Pulse Gas Control System accepts HP-nitrogen from the Power Section ASU and distributes cleaning pulses to the filter-reactor, as well as the other barrier filters (Items 9, 16, and 20) in this section.

#### Gas heat interchanger (Item 5):

The gas is cooled in a heat interchanger to 482°C (900°F). This gas is then mixed with recycled tail gas from the downstream, sulfur recovery process, a flow of about 10% of the gas stream.

#### Bulk desulfurization:

The gas now enters the "humid-gas" bulk-desulfurization process where its total sulfur content is reduced to less than 50 ppmv, about a 99.5% reduction of  $H_2S$ , COS,  $CS_2$ , Sx, and  $SO_2$  contained in the gas, with significant hydrolysis of  $CS_2$ , Sx and  $SO_2$  to  $H_2S$  also expected. This meets the entire desulfurization needs for the Current Standards case, so long as no other sulfur release points exist in the power plant.

Any, of a number of bulk-desulfurization processes that are under development using a regenerative desulfurization sorbent, effective above the gas dew point, could be applied. In this evaluation, the well-developed, zinc titanate-based, transport reactor desulfurization process is applied. This selection results in the need for about a 482°C (900°F) inlet temperature. The zinc titanate sorbent is assumed to have a Zn/Ti mole ratio of 1.0, and to operate with a net, sorbent makeup stoichiometric molar feed ratio of 0.004 Zn/S, 45 kg/hr (98 lb/hr) provided by the sorbent feed system, Item 7.

The gas passes through a transport reactor (Item 6) of circulating zinc titanate sorbent, producing a bulk-desulfurized gas having total sulfur content of about 40 ppmv and containing some entrained sorbent particles that escape the transport reactor disengaging section. The partially-sulfided sorbent particles circulating to the desulfurizer leg have a molar ratio for ZnO/S of about 174. The sulfided sorbent particles circulate to the parallel, entrained regenerator vessel (Item 8) where air contacting generates an SO<sub>2</sub> acid gas and regenerated zinc titanate sorbent. Compressed air is provided by compressor Item 12, and a fired heater (Item 11) is also provided for preheating the regenerator air. Nitrogen fluffing gas and nitrogen purging of the transport legs between the gasifier and regenerator are used, this compressed nitrogen coming from the Power Section. The regenerator acid gas, at about 1350°F, passes through a relatively small barrier filter (Item 9) to separate its entrained sorbent particles. The entrained sorbent particles captured in this filter are cooled and drained back to the standleg of sorbent flowing back to the desulfurizer vessel, or may be drained into the bulk desulfurized fuel gas exiting the vessel.

The bulk-desulfurized gas leaves the process at about 549°C (1020°F) carrying all of the sorbent lost by attrition and elutriation from the bulk-desulfurization process. The bulk desulfurized gas is at 552°C (1026°F) and 2365 kPa (343 psia), and its composition is listing in Table 5.5. Note that it contains significant particulate in the form of zinc-titanate sorbent particles. The relatively high nitrogen content in the gas results largely from the recycled sulfur recovery tail gas. The acid gas generated has the composition listed in Table 5.6.

	Major constituents (vol%)
H <sub>2</sub>	22.94
CH <sub>4</sub>	0.28
СО	36.68
CO <sub>2</sub>	10.27
H <sub>2</sub> O	19.52
N <sub>2</sub>	10.10
Ar	0.19
Total	99.98
	Major contaminants (ppmv)
$H_2S$	39.98
COS	2.85
HCl	3.5
NH <sub>3</sub>	158
HCN	19
Hg (ppbv)	2.5
particulate (ppmw)	265

 Table 5.5 - Bulk Desulfurized Gas Composition - Current Standards

Table 5.6 - Acid Gas Composition – Novel Gas Cleaning with Current Standards

	Major constituents (vol%)		
$N_2$	83.77		
Ar	0.96		
$CO_2$	0.03		
H <sub>2</sub> O	1.03		
SO <sub>2</sub>	14.20		
Total	99.99		

### Gas cooling and barrier filter:

The bulk desulfurized gas is cooled in two process heat exchangers (13 and 18) to a temperature of no lower than 204°C (400°F), suitable for mercury removal in the Mercury Removal Section. The first is a recuperative heat interchanger (Item 13) used to reheat the clean fuel gas from 204°C (400°F) to 404°C (760°F) and passes the gas and entrained sorbent particles into a barrier filter-reactor (Item 16). The entrained sorbent is collected on the filter elements, allowing additional sulfur removal. The sorbent particles may be recycled to the Bulk Desulfurizer regenerator, disposed of, or sent to the gasifier to be incorporated into the slag. The second is a BFW-cooled heat exchanger that completes the fuel gas cooling and preheats BFW. The clean fuel gas then passes to recuperative heat interchanger (Item 5) where it is reheated to 507°C (945°F).

### Soot-blower gas and Sulfur Recovery fuel gas:

A small portion of the bulk-desulfurized gas is separated, cooled and compressed at this point for use as soot-blower gas in the slagging heat exchangers. About 2.9% of this gas stream is separated at this point to be used in the Sulfur Recovery process.

## Mercury sorbent feed system:

Two mercury removal process schemes are considered, since this aspect of the plant is uncertain at this time: 1) once-through operation with powdered mercury sorbent injection into a filter-reactor, and

2) regenerative operation with powdered mercury sorbent injection into a filter-reactor. Only the oncethrough process is shown in Figure 5.3.

A TDA sorbent for mercury removal, and identification of its associated best operating conditions, have been established in the PDU test program, but it is assumed for the process evaluation that potential adsorbents (e.g., a zeolite) or sorbents operable at greater than 204°C (400°F) exist. Specific operating conditions are based on the mercury sorbent work by TDA Research, Inc. (Alptekin et al., 2003) and the PDU testing in this program. Based on this background, a mercury sorbent might effectively adsorb elemental mercury in syngas at temperatures as high as 260°C (500°F), with a gas residence time of about 0.1 second, and achieve a sorbent uptake of about 0.04 wt% (grams mercury per gram sorbent). The hypothetical sorbent could be regenerated by heating it to 288-299°C (550-570°F) in an inert gas if regenerative operation is desired.

The mercury sorbent is injected into the gas by feed system (Item 19). The assumed feed rate is 6.8 kg/hr (15 lb/hr), which represents a mass ratio of adsorbent to mercury vapor of greater than 1000.

## Mercury removal filter-reactor:

The cooled gas passes into a ceramic, or metal candle barrier filter (Item 20) to conduct mercury removal. Optionally, if the barrier filter (Item 16) was eliminated, the mercury filter-reactor would also collect the bulk desulfurization sorbent contained in the gas, and any equipment corrosion particulate that might be present in the gas from upstream sources. The mercury sorbent and other particulate is drained from the filter and is depressurized (Item 21) to be stored for disposal, probably as a hazardous solid waste, or regenerated if only mercury sorbent is present.

## Regenerative Operation of Mercury Sorbent:

If regenerative sorbent operation where used, the mercury sorbent drained from the filter would be contacted with steam or warmed nitrogen in an entrained reactor segment, heating the sorbent to about 288°C (550°F) and driving off the captured mercury. This product stream would be separated in a small barrier filter, and the separated sorbent particles would be pneumatically conveyed back to the mercury removal filter-reactor. The water vapor and mercury vapor stream from the filter would be cooled and condensed, and the very small amount of mercury separated from the liquid water would be stored as a by-product. Waste sorbent produced in the process would be low in mercury content and may be disposed directly as a non-hazardous solid waste, or may be fed to the gasifier to be incorporated into the plant's slag by-product.

The fates of contaminants in the Gas Cleaning Section are summarized in Table 5.7.

Table 5.7 – Fate of Contaminants in Fuel Gas Cleaning Section – Novel Gas Cleaning with
Current Standards

Contaminant	Removal	Contaminant conversions
	(% of inlet to Section)	
sulfur species	99.54	Partial hydrolysis
		Combustion to SOx
		Sorbent fixation
halides	99	Sorbent fixation
ammonia	75	Catalytic partial decomposition to nitrogen
hydrogen cyanide	0	
mercury	90	Sorbent fixation

### **Sulfur Recovery Section**

Figure 5.4 shows the process flow diagram for the Sulfur Recovery Section of the IGCC power plant. Table 5.8 lists characteristics of major process streams. The acid gas from the bulk desulfurizer

regenerator contains about 14 vol% SO<sub>2</sub>, with negligible oxygen content, and the remaining components being largely nitrogen with a little CO<sub>2</sub>. This acid gas is first expanded (Item 1) to about 193 kPa (28 psia), cooling the gas to about 353°C (668°F) and generating a small amount of electrical power. Modern Claus technology allows relatively low H<sub>2</sub>S content, in the range of 5 - 10 vol%, to be efficiently and economically processed to recover sulfur (Bruijn et al., 2003).

The acid gas is then mixed with a portion of bulk desulfurized gas and is catalytically reacted to hydrogenate (Item 2) an appropriate portion of the acid gas  $SO_2$  to  $H_2S$  for the Claus reaction. This conversion requires a substantial fraction (about 2.9%) of the desulfurized gas and results in some degradation in the overall performance of the power plant. The gas is then cooled in a boiler (Item 3) to generate IP-steam, followed by a boiler-sulfur condenser (Item 4) generating LP-steam. Any elemental sulfur contained in the product acid gas is separated before the gas enters the first Claus reactor (Item 5). The remaining steps of the process are very similar to those described for the conventional gas cleaning sulfur recovery process: three stages of gas preheat, Claus reactors and sulfur condensers. If a sulfur product other than elemental sulfur were selected as the final disposition for the captured sulfur, such as CaSO<sub>4</sub> or sulfuric acid, the Novel Gas Clean process would show performance advantages over the conventional gas cleaning technology even greater than shown in this evaluation (Wasaka, 2003).

The sulfur recovery process recovers about 95.7% of the sulfur content of the acid gas. The collected sulfur streams are combined, and stored for treatment and marketing as a by-product. The tail gas contains a considerable content of sulfur species ( $H_2S$  and  $SO_2$ ), and the only way the power plant can achieve its overall 99.5% sulfur removal goal is for this tail gas to be recompressed and recycled to the bulk desulfurizer. After cooling, compression and condensate removal, the tail gas recycled to the bulk desulfurizer has the composition reported in Table 5.9.

Stream name	Acid Gas to	Clean fuel gas	Sulfur	Tail gas
	Sulfur		product	recycle
	Recovery			
Molar flow, kmole/hr	749	361	113	858
Mass flow, kg/hr	24,840	7,847	3,466	26,758
Volumetric flow, m <sup>3</sup> /hr	2.752	650	2.0	1420
Temperature, °C	742	204	137	217
Pressure, kPa	2317	2207	163	2482
Enthalpy, MJ/hr	-15,610	-44,358	-12,557	-64,081
Molecular wt	33.1	21.7	30.7	31.2

Table 5.8 – Stream Characteristics for Sulfur Recovery Section – Current Standards

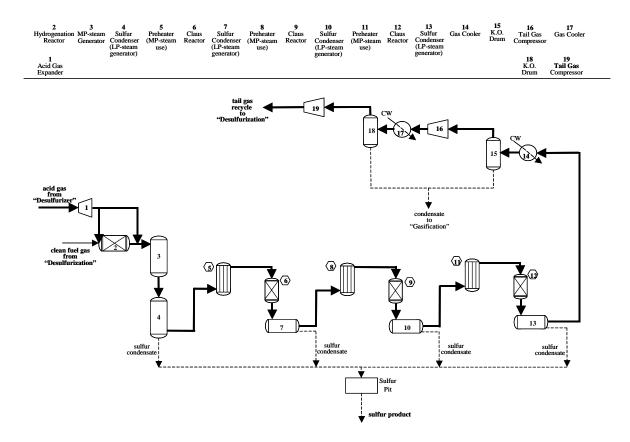


Figure 5.4 - Sulfur Recovery Section – Novel Gas Cleaning

	Major constituents (vol%)
H <sub>2</sub>	0.12
CH <sub>4</sub>	0.12
CO	0.17
$CO_2$	19.63
H <sub>2</sub> O	1.09
$N_2$	77.43
Ar	0.91
Total	99.47
	Major contaminants (ppmv)
$H_2S$	3665
$SO_2$	1648
COS	1
$CS_2$	0
NH <sub>3</sub>	46
HCN	8

Table 5.9 - Recycle Tail Gas Composition – Novel Gas Cleaning with Current Standards

The fate of the contaminants within the Sulfur Recovery section are listed in Table 5.10.

Contaminant	Removal (% of inlet to Section)	Contaminant conversions
sulfur species	95.72	Claus conversion to elemental sulfur Hydrolysis to reduced forms
halides	NA	
ammonia	30.0	Furnace decomposition
hydrogen cyanide	NA	
mercury	NA	

## Table 5.10 – Fate of Contaminants in Sulfur Recovery Section – Novel Gas Cleaning with Current Standards

## **Power Section**

The Power Section process flow diagram is shown in Figure 5.5. Table 5.11 lists characteristics of major streams. An oxygen stream with 95% purity, is generated by conventional, pressurized, cryogenic air separation (ASU). A relatively low-purity  $N_2$  stream is also produced and compressed (Item 21) that is used for clean fuel gas dilution. A smaller stream of high-purity  $N_2$  (99.9%) is also produced and compressed (Item 22) that is used in the gas cleaning process for solids pressurization, stripping, purging, pneumatic transport, and filter pulse cleaning. Low-purity nitrogen is humidified (Item 25) to about 15 vol% water vapor using low-grade heat sources (3, 23), and the nitrogen stream is mixed with the clean fuel gas before the mixture is distributed to the gas turbine combustors (Item 6).

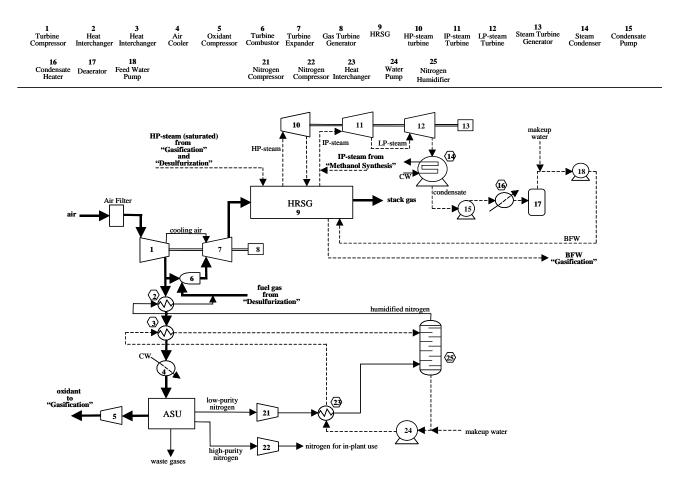
The turbine combustors operate with an outlet temperature of about 1521°C (2770°F), and with the peak flame temperature of less than 1649°C (3000°F), the NOx emission is expected to be less than 15 ppmv (dry, corrected to 15% oxygen). These are special burners designed for the efficient combustion of low heating-value fuel gases with low NOx production. They are expected to use a staged combustion design that promotes the decomposition of ammonia with less than 5% ammonia conversion to NOx. The turbine expansion gas has a temperature of 619°C (1147°F). The expansion gas passes through the heat recovery steam generator (HRSG), generating a superheated, high-pressure steam flow of 370,810 kg/hr (817,500 lb/hr). The stack gas from the power plant has a temperature of 121°C (250°F) and a composition listed in Table 5.12.

Table 5.11 -	- Stream (	Characteristics	for Power	Section –	Current	Standards
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Stream name	Air to gas	Gasifier	Low-purity	Makeup	Fuel gas to	Stack gas
	turbine	oxidant	nitrogen	water	gas turbine	
Molar flow, kmole/hr	53,621	2,857	9,946	1,863	11,491	60,137
Mass flow, kg/hr	1,547,310	91,026	280,171	33,566	249,665	1,725,182
Volumetric flow, m <sup>3</sup> /hr	1,279,105	2,343	31,298	34	34,189	1,944,040
Temperature, °C	15	81	27	15	507	121
Pressure, kPa	100	3585	793	2068	2,193	101
Enthalpy MJ/hr	-1.52E+05	4,221	101	-5.34E+05	-1.30E+06	-3.71E+06
Molecular wt	28.9	31.9	28.2	18.0	21.7	28.7

 Table 5.12 - Stack Gas Composition – Novel Gas Cleaning with Current Standards

	Major constituents (vol%)
O <sub>2</sub>	8.17
$CO_2$	9.04
H <sub>2</sub> O	11.99
$N_2$	69.95
Ar	0.84
Total	99.99
	Major contaminants
SO <sub>2</sub> (ppmv)	8.2
NOx (ppmv)	15
Mercury (ppbv)	0.05
Halides (ppmv)	0.67



**Figure 5.5 - Power Section** 

The fate of the contaminants within the Power section are listed in Table 5.13.

Table 5.13 – Fate of Contaminants in Power Section – Novel Gas Cleaning - Current Standards

Contaminant	Removal (% of inlet to Section)	Contaminant form conversion
sulfur species	0	Oxidation to SOx
halides	NA	
ammonia	0	Partial oxidation conversion to NOx
hydrogen cyanide	NA	
mercury	0	Partial conversion to oxidized forms

# 5.3 IGCC WITH NOVEL GAS CLEANING TO MEET FUTURE STANDARDS

The IGCC power plants using the Novel Gas Cleaning technology to achieve Future Standards that approach the emissions performance of natural gas-fired power plants has the following targets:

- SOx: 99.98% coal sulfur removal "net" for entire power plant,
- NOx: 5 ppmv (corrected to 15% oxygen, dry) at the plant stack,
- particulate: 0.1 ppmw in the fuel gas,
- mercury: 95% coal mercury removal,

- Power Island contaminants:
  - halides: 5 ppmv in the fuel gas (after humidification, before nitrogen dilution)
  - metals (Ni, Fe): formation of metal carbonyls unlikely in the process
  - particulate: 0.1 ppmw in the fuel gas.

The gas cleaning system description for this IGCC application is almost the same as that just described for Novel Gas Cleaning for Current Standards, except for the additional of a Sulfur Polishing Filter-Reactor to the Desulfurization Section, and resulting changes to the clean fuel gas reheat temperatures.

### **Gasification Section**

Figure 5.2 shows the process flow diagram constructed for the Gasification Section using the Novel Gas Cleaning process. The process diagram and the stream flows and conditions are identical to the Gasification Section process flow diagram for the Current Standards case.

## **Fuel Gas Cleaning Section**

Figure 5.6 shows the process flow diagram for the Fuel Gas Cleaning Section. It is identical to Figure 5.3, for the Current Standards case, except that a Sulfur Polishing Filter-Reactor (Item 16) and Polishing Sorbent Feed System (Item 15), and Sorbent Waste Removal System (Item 17) have been inserted after the bulk desulfurizer for additional sulfur removal.

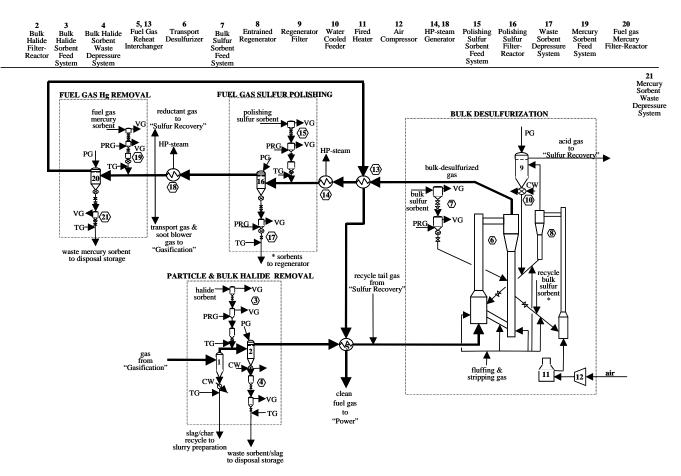


Figure 5.6 - Fuel Gas Cleaning Section for Novel Gas Cleaning - Future Standards

#### Gas heat interchanger (Item 5):

The gas is cooled in a recuperative heat interchanger to 482°C (900°F). This gas is then mixed with recycled tail gas from the downstream, sulfur recovery process, a flow of about 11% of the main gas stream.

#### Bulk desulfurization:

The gas now enters humid-gas, bulk-desulfurization process where its total sulfur content is reduced to less than 50 ppmv, about a 99.5% reduction of H<sub>2</sub>S, COS, CS<sub>2</sub>, Sx, and SO<sub>2</sub> contained in the gas, with significant hydrolysis of CS<sub>2</sub>, Sx and SO<sub>2</sub> to H<sub>2</sub>S also expected. The zinc titanate sorbent is assumed to have a Zn/Ti mole ratio of 1.0, and to operate with a net, sorbent makeup stoichiometric molar feed ratio of 0.0027 Zn/S provided by the sorbent feed system. The gas passes through a transport reactor (Item 6) of circulating zinc titanate sorbent, producing a bulk-desulfurized gas having total sulfur content of about 40 ppmv and containing some entrained sorbent particles that escape the transport reactor disengaging section. The partially-sulfided sorbent particles circulating in to the desulfurizer leg have a molar ratio for ZnO/S of about 219. The sulfided sorbent particles circulate to the parallel, entrained regenerator vessel (Item 8) where air contacting generates an  $SO_2$  acid gas and regenerated zinc titanate sorbent. Compressed air is provided by compressor, and a fired heater (Item 11) is also provided for preheating the regenerator air. Nitrogen fluffing gas and nitrogen purging of the transport legs between the gasifier and regenerator are used, this compressed nitrogen coming from the Power Section. The regenerator acid gas, at about 741°C (1365°F), passes through a relatively small barrier filter (Item 9) to separate its entrained sorbent particles. The entrained sorbent particles captured in this filter are cooled and sent back to the standleg of sorbent flowing back to the desulfurizer vessel, or may be drained into the bulk desulfurized fuel gas exiting the vessel. The bulk-desulfurized gas leaves the process at about 1025°F carrying all of the sorbent lost by attrition and elutriation from the bulk-desulfurization process. The bulk desulfurized gas has a flow of 267,826 kg/hr (590,459 lb/hr), at 553°C (1027°F) and 2365 kPa (343 psia), and its composition is listing in Table 5.14. Note that it contains significant particulate in the form of zinc-titanate sorbent particles. The relatively high nitrogen content in the gas results largely from the sulfur recovery tail gas. The acid gas generated has the composition listed in Table 5.15. Its flow is 24,860 kg/hr (54,808 lb/hr) at 741°C (1365°F) and 2327 kPa (336 psia).

#### Sulfur Polishing filter-reactor:

Polishing sulfur sorbent particles, also zinc titanate-type, are injected (item 15) into the gas and, combined with the entrained sorbent particles from the bulk-desulfurization process, the mixture enters a filter-reactor (Item 16) and results in additional 96% sulfur removal down to a level of total sulfur less than 2 ppmv. The sorbent makeup rate uses a stoichiometric ratio of 0.2 Zn/S in the regenerative operation. The partially-sulfided sorbent particles carried with the gas have a molar ratio for ZnO/S of about 3.3. The filter separates the entrained bulk-sorbent particles and the polishing sorbent particles from the gas. The collected sorbent particulate is drained from the filter (Item 17) and is pneumatically fed back to the bulk desulfurization process regenerator. The small amount of waste sorbent ultimately drained from this system is either disposed directly or is fed to the gasifier to be incorporated into the plant slag by-product.

#### Gas cooling:

The bulk desulfurized gas is cooled in process heat exchangers (Item 13) to 900°F and in cooler Item 18 to a minimum temperature of about 400°F, suitable for mercury removal in the Mercury Removal Section. The Hg-cleaned fuel gas passes through heat interchangers (Item 13) and to reheat the clean fuel gas from 400°F to 761°F. Item 18 is a BFW-cooled heat exchanger that completes the gas cooling and preheats BFW. The clean fuel gas then passes to heat interchanger (Item 5) where it is reheated to 946°F.

Soot-blower gas is separated, cooled and compressed at this point for use as soot-blower gas in the slagging heat exchangers. About 2.9% of this gas stream is separated at this point to be used in the sulfur recovery process.

## Mercury Removal:

The mercury removal process is similar to the previous description, but is designed and operated for 95% mercury removal. Two mercury removal process schemes almost identical to those with the Current Standards case are considered: 1) once-through operation with powdered mercury sorbent injection into a filter-reactor, and 2) regenerative operation with powdered mercury sorbent injection into a filter-reactor.

The mercury sorbent is injected into the gas by feed system 19. The assumed feed rate is 15 lb/hr, the same as was assumed in the Current Standards case for 90% mercury removal. The cooled gas passes into a ceramic, or metal candle filter-reactor (Item 20) to conduct mercury removal. This barrier filter is injected with a small flow of a high-temperature, mercury sorbent to remove 95% of the elemental mercury in the gas. The filter also collects any equipment-corrosion particulate that might be present in the gas from upstream sources, and the sorbent may also capture metal carbonyls, should they be generated. The mercury sorbent is drained from the filter and is depressurized (Item 17) to be disposal as a hazardous solid waste.

	Major constituents (vol%)
H <sub>2</sub>	22.93
CH <sub>4</sub>	0.28
СО	36.67
CO <sub>2</sub>	10.27
H <sub>2</sub> O	19.51
N <sub>2</sub>	10.10
Ar	0.19
Total	99.95
	Major contaminants (ppmv)
$H_2S$	39.96
COS	2.85
HCl	3.5
NH <sub>3</sub>	158
HCN	19
Hg (ppbv)	2.5
particulate (ppmw)	1284

Table 5.14 - Bulk Desulfurized Gas Composition - Future Standards

Table 5.15 - Acid	Gas Composition -	- Novel Gas	Cleaning for	<b>Future Standards</b>

	Major constituents (vol%)
N <sub>2</sub>	83.77
Ar	0.96
CO <sub>2</sub>	0.03
H <sub>2</sub> O	1.03
$SO_2$	14.20
Total	99.99

The polished fuel gas composition following mercury removal is listed in Table 5.16.

	Major constituents (vol%)
H <sub>2</sub>	22.94
CH <sub>4</sub>	0.28
СО	36.68
$CO_2$	10.27
H <sub>2</sub> O	19.52
N <sub>2</sub>	10.11
Ar	0.19
Total	99.99
	Major contaminants (ppmv)
$H_2S$	1.6
COS	0.1
HC1	3.5
NH <sub>3</sub>	158
HCN	19
Hg (ppbv)	2.5
particulate (ppmw)	0.1

Table 5.16 - Polished Fuel Gas Composition – Novel Gas Cleaning for Future Standards

The fates of the contaminants estimated for the Fuel Gas leaning Section are listed in Table 5.17.

Table 5.17         – Fate of Contaminants in Fuel Gas Cleaning Section – Novel Gas Cleaning for Future
Standards

Contaminant	Removal	Contaminant conversions
	(% of inlet to Section)	
sulfur species	99.98	Partial hydrolysis
		Combustion to SOx
		Sorbent fixation
halides	99	Sorbent fixation
ammonia	75	Catalytic partial decomposition to nitrogen
hydrogen cyanide	0	
mercury	95	Sorbent fixation

Comments on the Mercury Removal process:

The performance of the filter-reactors with injected sorbents have not yet been experimentally established, so the process evaluation is speculative. In particular, the type of mercury sorbent, the characteristics of the mercury removal process, and the performance of the mercury removal process are all highly speculative at this time. The evaluation identifies the process' acceptable range of operating conditions, required range of performance, and potentially acceptable operational modes:

• mercury removal should operate as hot as about 204-316°C (400-600°F), removing 90-95% of the mercury,

- the type of mercury sorbent has not been established, and a TDA sorbent has been used in this evaluation, and it could be either a once-through or a regenerative sorbent -- it is expected that it may be advantageous for it to be a regenerative adsorbent, but this is not a necessity for process feasibility,
- the mercury removal process should be a continuous process -- it is expected that a continuous process will have advantages over a batch process with respect to power plant availability and performance,
- the selected mercury sorbent must not result in the release of any contaminants, such as sulfur, to the cleaned fuel gas that will exceed the emission requirements,
- the mercury adsorbent, if regenerative, might be regenerated by heating it in an available, clean gas or vapor stream, such as nitrogen or steam, to a temperature of no greater than 343°C (650°F), with liquid mercury being subsequently condensed and separated,
- the mercury removal step provides the final, clean fuel gas that goes to the gas turbine combustors, and it should have the capability of also handling upstream equipment corrosion particulate removal,
- the minimum operating temperature for the mercury removal stage is about 204°C (400°F), based on vapor condensation -- if lower operating temperatures are desired, water vapor will be condensed from the fuel gas.

## **Sulfur Recovery Section**

Figure 5.6, the process flow diagram for the Sulfur Recovery Section of the IGCC power plant, is almost identical for both the Current Standards and Future Standards cases. The acid gas from the bulk desulfurizer regenerator contains about 14 vol%  $SO_2$ , with very low oxygen content, and the remaining components being largely nitrogen with a little  $CO_2$ .

The sulfur recovery process recovers about 95.7% of the sulfur content of the acid gas. The collected sulfur streams are combined and the sulfur is stored for treatment and marketing as a by-product. The tail gas, at 29,247 kg/hr (64,478 lb/hr), contains a considerable content of sulfur species ( $H_2S$  and  $SO_2$ ), and the only way the power plant can achieve its overall 99.85% sulfur removal goal is for this tail gas to be recompressed and recycled to the bulk desulfurizer. After cooling and compression and condensate removal, the tail gas recycled to the bulk desulfurizer has the composition reported in Table 5.18.

	Major constituents (vol%)
$H_2$	0.12
$CH_4$	0.12
CO	0.17
$CO_2$	19.63
$H_2O$	1.09
$N_2$	77.43
Ar	0.91
Total	99.47
	Major contaminants (ppmv)
$H_2S$	3675
$SO_2$	1648
COS	0
$CS_2$	0
NH <sub>3</sub>	6
HCN	8

# Table 5.18 - Recycle Tail Gas Composition – Novel Gas Cleaning for Future Standards

## **Power Section**

The Power Section process flow diagram is the same as that shown for the Current Standards case in Figure 5.7. An oxygen stream at 91,026 kg/hr (200,679 lb/h)r, with 95% purity, is generated by conventional, pressurized, cryogenic air separation (ASU). A relatively low-purity N<sub>2</sub> stream is also produced that is used for clean fuel gas dilution. A smaller stream of high-purity N<sub>2</sub> (99.9%) is also produced that is used in the gas cleaning process for solids pressurization, stripping, purging, pneumatic transport, and filter pulse cleaning. Low-purity nitrogen is humidified to about 15 vol% water vapor using low-grade heat sources, and the nitrogen stream at 313,736 kg/hr (691,672 lb/hr) is mixed with the clean fuel gas before the mixture is distributed to the gas turbine combustors. The turbine combustors, advanced, catalytic, and/or diffusion flame burners that promote the decomposition of the remaining ammonia in the fuel gas with less than 5% conversion to NOx, operate with an outlet temperature of about 1521°C (2770°F), and with the peak flame temperature of less than 1649°C (3000°F), the NOx emission is expected to be less than 5 ppmv (dry, corrected to 15% oxygen).

The turbine expansion gas has a mass flow of 1,724,957 kg/hr (3,802,899 lb/hr) and a temperature of 619°C (1147°F). The expansion gas passes through the heat recovery steam generator (HRSG), generating a superheated, high-pressure steam flow of 370,810 kg/hr (817,500 lb/hr). The stack gas from the power plant has a temperature of 121°C (250°F) and a composition listed in Table 5.19.

	Major constituents (vol%)	
O <sub>2</sub>	8.17	
CO <sub>2</sub>	9.04	
H <sub>2</sub> O	11.99	
$N_2$	69.95	
Ar	0.84	
Total	100.00	
	Major contaminants	
$SO_2(ppmv)$	0.3	
NOx (ppmv)	5	
halides	0.68	
mercury (ppbv)	0.025	

 Table 5.19 - Stack Gas Composition – Novel Gas Cleaning for Future Standards

### 5.4 DESCRIPTION AND PERFORMANCE OF FILTER-REACTORS

Designs and estimates of performance of the three filter-reactors used in the IGCC Novel Gas Cleaning Process have been developed. The three filter-reactors are 1) the Halide Filter-Reactor, 2) the Sulfur Polishing Filter-Reactor, and 3) the Mercury Removal Filter-Reactor. The designs were developed based on the requirements for the filter-reactor to perform as an effective particle filter and as an effective chemical reactor for contaminant removal. The barrier filter design aspects were based on commercial barrier filter design methods and supply experience for units in the required capacity range. The chemical reactor performance estimates were based on numerical modeling of transient filter cake contaminant removal using available reaction kinetics information for the proposed types of sorbents. The reaction kinetic behavior information is significantly uncertain at this time and these predicted results are considered preliminary. They are applied for the assessment of equipment performance feasibility and for program planning purposes. These results will be refined when PDU testing of sub-scale filter-reactors has been completed and the test data has been analyzed to extract contaminant removal performance parameters. The results reported below provide the basic filter-reactor design dimensions, weights, filter performance, and contaminant removal performance estimates. Preliminary conclusion are also extracted and reported.

### **Halide Filter-Reactor**

The Halide Filter-Reactor has several performance objectives: 1) efficiently removal char/slag particulate contained in the raw fuel gas, 2) remove sufficient HCl from the fuel gas to meet the assumed plant requirement of 5 ppmv HCl in the fuel gas to the gas turbine, and 3) induce some catalytic decomposition of ammonia contained in the raw fuel gas to assist in the final control of NOx emitted from the plant. The raw fuel gas is cooled sufficiently so that the gasifier particulate will be in the form of solidified slag and char, and is also pre-cleaned by particle removal through an efficient cyclone to limit the slag/char loading. HCl is removed by injecting a particulate sorbent into the filter that will react with HCl in the entrained zone of the filter as well as in the filter cake accumulated on the filter elements. Decomposition of ammonia is inherently induced within the filter-reactor by the catalytic activity of the high-alloy materials used in parts of the filter internals. It is also possible to pack regions of the filter-reactor vessel with similar catalytic alloy-materials in forms such as wire mesh to contact the fuel gas for additional ammonia decomposition.

Table 5.20 lists the sorbent assumptions and expected performance for the filter-reactor. Nacholite, pneumatically fed as -325 mesh particles, is used as the HCl sorbent, and several other cheap mineral forms high in sodium content, such as Trona, could also be used. It is used in a once-through operation, with the reacted Nacholite sorbent to be disposed as a non-hazardous solid waste. The total particulate loading of char and sorbent in the gas to the filter-reactor is about 9,800 ppmw, a typical-to-low level of loading in prior barrier filter test experience. HCl removal of about 99% is needed to meet the IGCC plant requirement, and it is assumed that ammonia decomposition of about 75% is possible based on literature reports on this subject (see, for example, work reported by: S. A. Qader, et al., Energy and Environmental Technology Corporation, "Decomposition of Ammonia. in IGCC Fuel Gas Streams," Proceedings of Advanced Coal-fired Power Systems Review Meeting, Morgantown, WV, July 1996, CONF-960757--24 ; and W. Wang et al., Lund University, "Reduction of Ammonia and Tar in Pressurized Biomass Gasification," International Symposium on Gas Cleaning at High Temperatures, 2002). The barrier filter has been shown to reliably achieve outlet particle loadings less than 0.1 ppmw in prior gasifier testing (Southern Company Services, PSDF Test Reports).

Sorbent type	Nacholite powder
Sorbent feed size	-325 mesh
Sorbent feed rate, kg/hr (lb/hr)	2,019 (4,451)
Type of sorbent operation	Once-through
Na/Cl molar feed ratio	4.0
Inlet gas total particle loading, ppmw	9.817
HCl removal, %	99.1
NH <sub>3</sub> decomposition, %	75
Outlet particulate loading, ppmw	0.1

**Table 5.20 - Halide Filter-Reactor Sorbent Assumptions** 

Based on the values in Table 5.20, the filter-reactor inlet and outlet fuel gas stream characteristics in Table 5.21 have been computed. The change in fuel gas mass and molar flow rates across this filter-reactor are due to feed sorbent transport gas, pulse cleaning gas, and sorbent decomposition gas (water vapor).

	Inlet Stream	Outlet Stream
Fuel gas flow, kg/hr (lb/hr)	231,899 (511,253)	232,363 (512,276)
Fuel gas flow, kg-mole/hr (lb-	11,069 (24,403)	11,095 (24,460)
mole/hr)		
Fuel gas flow, m <sup>3</sup> /hr (ft <sup>3</sup> /min)	524 (18,516)	534 (18,845)
Temperature, °C (°F)	593 (1100)	586 (1087)
Pressure, kPa (psia)	2,551 (370)	2,496 (362)
Coal slag/char, ppmw	1,106	0
Total sulfur, ppmv	9253.9	9230.9
$H_2S$	8609	8588
$CS_2$	0.6	0.6
COS	634	632
S	7.4	7.4
SO <sub>2</sub>	2.9	2.9
HCl, ppmv	434	3.9
NH <sub>3</sub> , ppmv	687	171
HCN, ppmv	20	20
Hg, ppbv	2.7	2.8

Table 5.21 - Halide Filter-Reactor Inlet and Outlet Streams

To estimate the HCl removal performance of this filter-reactor, the reaction behavior of the entrained zone of the vessel and of the transient filter cakes were estimated by numerical modeling. The differential material conservation equations for the entrained zone were developed using the following assumptions:

- the entrained reaction zone acts as a steady-state, dilute reaction stage,
- fuel gas and entrained sorbent particles flow co-currently with limited slip velocity and little backmixing,
- the fuel gas and entrained sorbent particle contact time is assumed to be the average gas residence time of fuel gas in the vessel,
- the contaminant levels in the fuel gas are small enough that the total fuel gas molar flow, volumetric flow, and temperature do not change due to the removal reactions,
- gas-particle mass transfer rate and chemical reaction rate are both considered,
- the gas-particle mass transfer coefficient was estimated from available experimental correlations reported in the literature for entrained particle mass transfer based on calculated values of the particle-Reynolds Number and the particle-Schmidt Number.

Similarly, for the transient filter cake, the assumptions applied were:

- the fuel gas and sorbent particles arrive at the outer filter cake surface having their average entrained zone outlet compositions,
- the filter cake builds in depth as a function of time, assuming a constant mass flow rate of fuel gas and sorbent particles, until it is periodically pulse cleaned,
- the time-dependent and radial distance-dependent behavior of the fuel gas phase and the sorbent filter cake phase are described by a pair of differently material balance equations that are coupled through mass transfer and chemical reaction between the two phases,
- the contaminant levels in the fuel gas are small enough that the total fuel gas molar flow, volumetric flow, and temperature do not change across the filter cake due to the removal reactions,

- the gas-particle mass transfer coefficient was estimated from available experimental correlations reported in the literature for packed bed mass transfer based on calculated values for packed bed particle-Reynolds and Schmidt numbers.
- an empirical form of the gas contaminant-sorbent particle reaction rate was assumed:

Rate (moles/volume-second) = k  $A_s C_g (y - y_e) (1 - X)^m$ 

where k is the experimental rate constant,  $A_s$  is the sorbent particle external surface area per unit bed volume,  $C_g$  is the gas total molar density, y is the gas contaminant mole fraction in the bulk of the gas,  $y_e$  is the equilibrium content of contaminant that would exist in the gas, X is the mole fraction of the sorbent reactive species that has been converted, and m is an empirical rate factor.

Table 5.22 lists the key reaction parameters applied in the performance estimates for the Halide Filter-Reactor reaction performance. The HCl reaction rate parameters were taken from Siemens inhouse TGA test results for Trona and HCl reaction at conditions representative of the Halide Filter-Reactor. The same reaction rate parameters were applied in both the filter cake and the entrained zones.

Mean sorbent particle diameter, µm	20
Particle sphericity factor	0.7
Cake voidage, %	75
Dilute zone gas-particle mass transfer coefficient, m/s (ft/s)	3 (10)
Cake gas-particle mass transfer coefficient, m/s (ft/s)	0.6 (2)
HCl equilibrium content, ppmv	1
Reaction rate constant k, m/s (ft/s)	0.005 (0.015)
Empirical rate factor "m"	1.6

Table 5.22 - Halide Filter-Reactor HCl Removal Factors Estimated

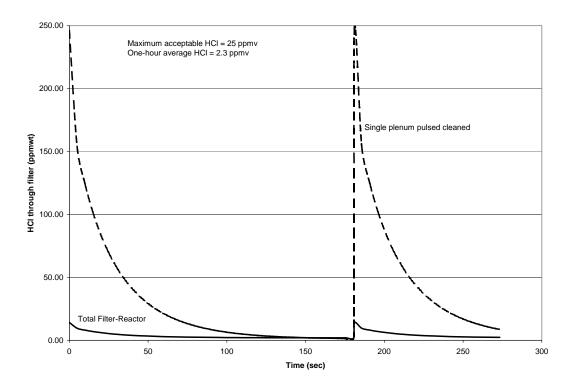
The estimated filter-reactor design and performance characteristics are listed in Table 5.23. The vessel design characteristics are typical of prior filter design, testing, and commercial supply experience. Twenty plenums are arranged in the filter vessel and are individually pulse cleaned at uniform intervals to maintain HCl removal control. The maximum vessel pressure drop occurs at the time of each plenum pulse cleaning event, and this pressure drop is reduced to the baseline pressure drop immediately following the pulse cleaning event. This is a shop fabricated pressure vessel that is transported by truck to the IGCC site.

About 30% of the HCl feed to the filter-reactor is expected to be removed in the dilute zone, with the remaining removed across the filter cake. Figure 5.7 illustrates the transient behavior of the filterreactor performance over a single pulse cleaning cycle of one plenum, showing the HCl emitted from the plenum being pulse cleaned, and the total Filter-Reactor HCl emission. The item in Table 5.23 listed as "average HCl from working plenums" is the level of HCl emitted from the 19 plenums that are not pulse cleaned at a given pulse cleaning event, and this level is estimated to be very near the equilibrium HCl content for the given reaction rate factors applied, but a conservatively-higher value has been assumed. The term "highest HCl from filter at pulse event" is the HCl content in the fuel gas passing through the filter-reactor immediately following a pulse cleaning event, and it is seen that this worst level is about 14 ppmv, slightly above the required HCl control level of 5 ppmv. The term "time to recover plenum to HCl control" is the time that it takes a plenum, once pulsed, to regain sufficient HCl removal to again control the HCl content of the gas to the average level of the filter plenums. Finally, the term " highest 1-hour average HCl from filter" is the one-hour averaged HCl content in the fuel gas emitted from the filterreactor and is representative of an actual performance criteria that might be used for judging the filterreactor HCl control performance. It is seen that this one-hour average is expected to be below the HCl requirement of 5 ppmv.

Thus, it is expected that the Halide Filter-Reactor will satisfy the control level required for HCl in IGCC. Other major considerations that must be characterized in the test program for this filter-reactor are the impact of the gasifier slag/char on the filtration and reaction performance and the flow behavior of the particulate waste materials from the filter-reactor vessel drain nozzle.

Design	
face velocity, m/min (ft/min)	2.1 (6.9)
number parallel clusters	5
number plenums	20
total number candles	930
vessel outer diameter, m (ft)	3.5 (11.4)
vessel total height, m (ft)	17.7 (58)
vessel wall thickness, mm (inch)	44.5 (1.75)
vessel refractory thickness, mm (inch)	88.9 (3.5)
vessel weight with internals, tonne (ton)	142 (156)
Performance	
maximum vessel pressure drop, kPa (psi)	0.06 (8.4)
pulse cleaning frequency, 1/hr	1
solids storage capacity in vessel, hr	4
gas residence time in dilute zone, sec	5
HCl removal in the dilute zone, %	32
maximum cake thickness, mm (inch)	10.2 (0.4)
time between plenum pulses, min	3
average filter cake age, hr	0.5
average HCl from working plenums, ppmv	2
highest HCl from filter at pulse event, ppmv	14
time to recover plenum to HCl control, min	1
highest 1-hour average HCl from filter, ppmv	2.1

**Table 5.23 - Halide Filter-Reactor Characteristics** 



**Figure 5.7 - Halide Filter-Reactor HCl Removal Performance** 

### **Bulk Desulfurizer**

The Bulk Desulfurizer follows the Halide Filter-Reactor stage, reducing the total sulfur content of the fuel gas to less than 50 ppmv using a regenerative zinc titanate sorbent in a transport bed desulfurizer. Its discussion is included here to present the major assumptions applied and to display the changes in fuel gas composition that occur across this desulfurizer. The fuel gas from the Halide Filter-Reactor is cooled by indirect heat exchange to about 482°C (900°F), and a stream of recycled tail gas from the sulfur recovery process is mixed with the main fuel gas stream. The mixed gas stream then passes into the transport desulfurizer. The assumptions listed in Table 5.24 have been applied for material & energy balance development. Some sorbent is lost from the transport desulfurizer, and from the parallel entrained bed regenerator, primarily by sorbent physical attrition and elutriation from the disengaging section of these vessels. This lost sorbent is carried to the next process stage, the Sulfur Polishing Filter-Reactor, with the bulk desulfurized fuel gas. A high level of sulfur removal for H<sub>2</sub>S and COS is expected in the bulk desulfurizer. Effective hydrolysis of minor sulfur species (CS<sub>2</sub>, S<sub>x</sub>, SO<sub>2</sub>) is also expected. The catalysis of the water-gas-shift reaction is likely in the transport desulfurizer, but in this evaluation, zero water-gas-shift has been assumed.

Sorbent type	Zinc titanate
Sorbent feed size (mean)	100 micron
Feed rate, kg/hr (lb/hr)	45 (98)
Type of sorbent operation	regenerative
Zn/S makeup mole feed ratio	0.0027
ZnO/S molar circulation ratio	232
Source of sorbent losses	attrition and elutriation
Sulfur removal, %	99.5
Hydrolysis of $CS_2$ , $S_x$ , $SO_2$ , %	100
Extent of water gas shift, %	0

**Table 5.24 - Bulk Desulfurizer Sorbent Assumptions** 

Table 5.25 lists the bulk desulfurizer inlet and outlet fuel gas stream characteristics. A relatively large increase in flow occurs due to the recycled tail gas stream as well as fluffing gas and solids streams stripping gases. The relatively large particulate content in the outlet fuel gas stream is primarily attrited zinc titanate sorbent particulate. Uncertainty exists in the nature of this attrited sorbent (average level of sulfidation and its reactivity), but it was assumed to possess the average outlet sorbent sulfidation level in this evaluation.

	Inlet Stream	Outlet Stream
Fuel gas flow, kg/hr (lb/hr)	259,121 (571,267)	267,800 (590,401)
Fuel gas flor, kg-mole/hr (lb-	11,953 (26,351)	12,314 (27,148)
mole/hr)		
Fuel gas flow, m <sup>3</sup> /hr (ft <sup>3</sup> /min)	498 (17,594)	600 (21,198)
Temperature, °C (°F)	463 (866)	552 (1026)
Pressure, kPa (psia)	2,468 (358)	2,365 (343)
Particulate, ppmw	0	1277
Total sulfur, ppmv	8950	42.3
$H_2S$	8235	40.0
$CS_2$	0.5	0
COS	587	2.9
S	6.9	0
$SO_2$	121	0
HCl, ppmv	3.6	3.5
NH <sub>3</sub> , ppmv	162	158
HCN, ppmv	19	19
Hg, ppbv	2.5	2.4

 Table 5.25 - Bulk Desulfurizer Inlet and Outlet Streams

## **Sulfur Polishing Filter-Reactor**

The Sulfur Polishing Filter-Reactor has three functions: 1) capture sorbent-based particulate emitted from the bulk desulfurizer, 2) utilize this captured sorbent, plus fresh polishing sorbent to desulfurize the fuel gas, and 3) recycle the captured sorbent back to the bulk desulfurizer's regenerator vessel where this sorbent will be reactivated and used for further cycles of desulfurization.

Table 5.26 lists the sorbent assumptions applied for M&E balances and the estimated performance for the Sulfur Polishing Filter-Reactor. The same sorbent type as that used in the bulk desulfurizer would probably be fed to the Sulfur Polishing Filter-Reactor, except with a smaller mean particle size. The level of sulfur removal needed is about 96%, and the quantity of sulfur removed is about a factor of 20 smaller than the sulfur quantity removed in the bulk desulfurizer.

 Table 5.26 - Sulfur Polishing Filter-Reactor Sorbent Assumptions

Sorbent type	Zinc-titanate powder
Sorbent feed size	-325 mesh
Sorbent feed rate, kg/hr (lb/hr)	16 (36)
Type of sorbent operation	regenerative
Zn/S makeup mole feed ratio	0.2
ZnO/S circulation ratio	3.7
Inlet gas total particle loading, ppmw	1,343
Sulfur removal, %	96
Outlet particulate loading, ppmw	0.1

Table 5.27 shows that little change occurs to the characteristics of the fuel gas stream, other than to the sulfur species content, as it passed through the Sulfur Polishing Filter-Reactor.

	Inlet Stream	Outlet Stream
Fuel gas flow, kg/hr (lb/hr)	267,823 (590,459)	267,045 (588,736)
Fuel gas flor,	12,315 (27,149)	12,311 (27,142)
kg-mole/hr (lb-mole/hr)		
Fuel gas flow,	554 (19,565)	566 (19,994)
$m^{3}/min$ (ft <sup>3</sup> /min)		
Temperature, °C (°F)	482 (900)	482 (900)
Pressure, kPa (psia)	2344 (340)	2289 (332)
Particulate, ppmw	1277	0.1
Total sulfur, ppmv	42.8	1.71
$H_2S$	40.0	1.6
$CS_2$	0	0
COS	2.8	0.11
S	0	0
SO <sub>2</sub>	0	0
HCl, ppmv	3.5	3.5
NH <sub>3</sub> , ppmv	158	158
HCN, ppmv	19	19
Hg, ppbv	2.5	2.5

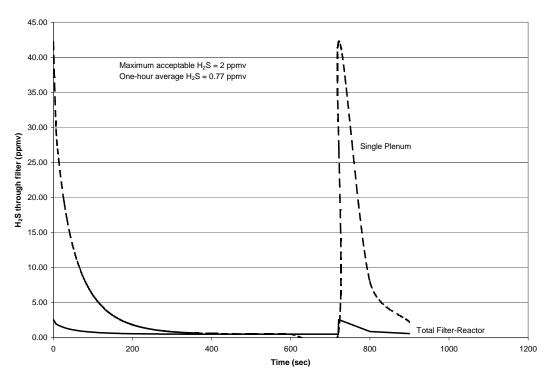
Table 5.27 - Sulfur Polishing Filter-Reactor Inlet and Outlet Streams

For estimating the sulfur removal performance of the Sulfur Polishing Filter-Reactor, the factors listed in Table 5.28 were estimated, being similar to those listed in Table 5.22 for the Halide Filter-Reactor, but are specific to H<sub>2</sub>S removal. The reaction factors reported in the table have been estimated from available packed bed test results for zinc-based sorbents found in the literature (for example, K. C. Kwon, Tuskegee University, "Kinetics of Hot Gas Desulfurization Sorbents for Transport Reactors," Annual Technical Process Report for FY1999 under contract DE-FG26-98FT40145--01, January 2000) as well as from Siemens in-house, pressurized TGA test results.

Mean sorbent particle diameter, µm	20
Particle sphericity factor	0.7
Cake voidage, %	75
Dilute zone gas-particle mass transfer coefficient, m/s (ft/s)	3 (10)
Cake gas-particle mass transfer coefficient, m/s (ft/s)	0.6 (2)
H <sub>2</sub> S equilibrium content, ppmv	0.5
Reaction rate constant k, m/s (ft/s)	0.024 (0.08)
Empirical rate factor "m"	2

Table 5.28 - Sulfur Polishing Filter-Reactor H<sub>2</sub>S Removal Factors Estimated

The Sulfur Polishing Filter-Reactor design described in Table 5.29 is very similar to the Halide Filter-Reactor design, and the analogous factors to those in Table 5.22 are reported. Only about 10% of the sulfur is expected to be removed in the entrained zone. Like the Halide Filter-Reactor, it has the potential to maintain the fuel gas sulfur content lower than is required by the IGCC application (less than 2 ppmv) on a one-hour time average basis, as is illustrated in Figure 5.8. In addition to characterizing the



reaction performance of the filter-reactor, the PDU testing will consider the filter cake behavior, pulse cleaning performance, and vessel hopper drainage performance.

## Figure 5.8 - Sulfur Polishing Filter-Reactor H<sub>2</sub>S Removal Performance

### **Mercury Removal Filter-Reactor**

The Mercury Removal Filter-Reactor also has multiple functions: 1) remove mercury from the fuel gas stream to a level of 95% removal (to about 0.125 ppbv Hg), 2) capture the injected mercury sorbent and transport it to a regenerator that will separate elemental mercury from the regenerated sorbent and will recycle the sorbent to the filter-reactor, and 3) remove any corrosion-based particulate from the fuel gas, as well as potentially remove other trace species from the fuel gas. The sorbent assumptions and performance estimates applied for M&E balances are listed in Table 5.30. The amount of circulating sorbent in the filter-reactor relative to the mercury flow is very high to maintain reliably-high mercury removal.

Design	
face velocity, m/min (ft/min)	2.2 (7.2)
number parallel clusters	5
number plenums	20
total number candles	930
vessel outer diameter, m (ft)	3.4 (11.3)
vessel total height, m (ft)	18 (59)
vessel wall thickness, mm (inch)	38 (1.5)
vessel refractory thickness, mm (inch)	70 (2.75)
vessel weight with internals, tonne (tons)	127 (140)
Performance	
maximum vessel pressure drop, kPa (psi)	0.05 (7.3)
pulse cleaning frequency, 1/hr	0.25
solids storage capacity in vessel, hr	17
gas residence time in dilute zone, sec	5
$H_2S$ removal in the dilute zone, %	12
maximum cake thickness, mm (inch)	6.4 (0.25)
time between plenum pulse, min	12
average filter cake age, hr	2
average $H_2S$ from working plenums, ppmv	1
highest H <sub>2</sub> S from filter at pulse event, ppmv	3
time to recover plenum to H <sub>2</sub> S control, min	4
highest 1-hour average H <sub>2</sub> S from filter, ppmv	1.1

 Table 5.29 - Sulfur Polishing Filter-Reactor Characteristics

 Table 5.30 - Mercury Removal Filter-Reactor Sorbent Assumptions

Sorbent type	Zeolite powder
Sorbent feed size	-325 mesh
Sorbent makeup feed rate, kg/hr (lb/hr)	7 (15)
Type of sorbent operation	Regenerative
Recycled sorbent rate, kg/hr (lb/hr)	82 (180)
Sorbent feed/Hg removed (mass ratio)	1,242
Sorbent circulation/Hg removed (mass ratio)	14,920
Inlet gas total particle loading, ppmw	273
Hg removal, %	95
Outlet particulate loading, ppmw	0

The inlet and outlet streams estimated for plant materials & energy balances for the Mercury Removal Filter-Reactor are listed in Table 5.31. Before the fuel gas reaches the Mercury Removal Filter-Reactor a portion of the fuel gas is extracted for soot-blower gas in the gasifier's radiant heat exchanger and for reducing gas in the sulfur recovery process, resulting in a reduced fuel gas flow compared to the other filter-reactors.

	Inlet Stream	Outlet Stream
Fuel gas flow, kg/hr (lb/hr)	249,665 (550,419)	249,672 (550,435)
Fuel gas flow, kg-mole/hr (lb-mole/hr)	11,491 (25,333)	11,491 (25,333)
Fuel gas flow, m <sup>3</sup> /min (ft <sup>3</sup> /min)	335 (11,827)	344 (12,164)
Temperature, °C (°F)	204 (400)	204 (400)
Pressure, kPa (psia)	2268 (329)	2206 (320)
Particulate, ppmw	0	0
Total sulfur, ppmv	1.71	1.71
$H_2S$	1.6	1.6
$CS_2$	0	0
COS	0.11	0.11
S	0	0
SO <sub>2</sub>	0	0
HCl, ppmv	3.5	3.5
NH <sub>3</sub> , ppmv	158	158
HCN, ppmv	19	19
Hg, ppbv	2.5	0.13

Table 5.31 - Mercury Removal Filter-Reactor Inlet and Outlet Streams

The specific type of mercury removal sorbent that will be used has not been determined at this point in the program, but will be the subject of selection in upcoming laboratory testing. Estimates of generic adsorbent parameters listing in Table 5.32 are provided only for initial feasibility estimates and have been based on available literature information.

Mean sorbent particle diameter, µm	20
Particle sphericity factor	0.7
Cake voidage, %	75
Dilute zone gas-particle mass transfer coefficient, m/s (ft/s)	3 (10)
Cake gas-particle mass transfer coefficient, m/s (ft/s)	0.6 (2)
Hg equilibrium content, ppbv	0.001
Reaction rate constant k, m/s (ft/s)	0.003 (0.01)
Empirical rate factor "m"	1.5

Table 5.32 - Mercury Removal Filter-Reactor Hg Removal Factors Estimated

The physical characteristic of the Mercury Removal Filter-Reactor listed in Table 5.33 are wellfounded, although the mercury removal performance is uncertain at this time. The filter-reactor vessel and internals are considerably cheaper than those of the Halide Filter-Reactor and the Sulfur Polishing Filter-Reactor because of its smaller volumetric flow and lower temperature. No independent pulse cleaning skid is required for this filter-reactor because of its infrequent pulsing, and it utilizes the equipment in the Sulfur Polishing Filter-Reactor pulse control skid. It is estimated that very little entrained zone mercury removal will occur due to the low mercury content in the fuel gas. The cleaning pulses are very infrequent in this filter-reactor and the removal performance is expected to be adequate to meet the requirement for 0.13 ppbv.

Design	
face velocity, m/min (ft/min)	1.7 (5.5)
number parallel clusters	4
number plenums	16
total number candles	748
vessel outer diameter, m (ft)	3.0 (9.7)
vessel total height, m (ft)	16.8 (55)
vessel wall thickness, mm (inch)	35.1 (1.38)
vessel refractory thickness, mm (inch)	25.4 (1.0)
vessel weight with internals, tonne (tons)	92 (101)
Performance	
maximum vessel pressure drop, kPa (psi)	0.06 (9.1)
pulse cleaning frequency, 1/hr	0.03
solids storage capacity in vessel, hr	68
gas residence time in dilute zone, sec	6.5
Hg removal in the dilute zone, %	0
maximum cake thickness, mm (inch)	0.7
time between plenum pulses, min	120
average filter cake age, hr	17
average Hg from working plenums, ppbv	0.1
highest Hg from filter at pulse event, ppbv	0.17
time to recover plenum to Hg control, min	30
highest 1-hour average Hg from filter, ppbv	0.1

Table 5.33 - Mercury Removal Filter-Reactor Characteristics

### **Filter-Reactor Conclusions**

The evaluation has shown that the filter-reactor concept has the capability to be applied for the fuel gas cleaning functions required in an IGCC power plant with Future Standards. The three filter-reactors have equipment designs well within the prior experience of design, testing, and commercial supply, and their equipment costs are well-founded. The contaminant removal performances of the three filter-reactors are supported by the results of the PDU testing of prototype equipment, where process performance goals have been achieved under representative application conditions.

The evaluation also shows that the filter-reactor has several advantages over the more conventional packed bed reactor configuration for use in IGCC applications. The conventional packed bed reactor, if applied for similar halide, sulfur, and mercury removal applications might work well as a contaminant removal reactor, but is prone to the following practical issues:

• The packed bed reactor is a batch reactor that requires periodic isolation from the process, depressurization and cooling, sorbent removal, sorbent refill, repressurization, re-integration with the process gas and reheat. This may be done with one, or more, parallel reactor vessels that are periodically switched in operation from adsorption to regeneration and/or recharge using switching valves and bypass piping.

- The packed bed reactor for large-scale fuel gas flows is not a simple design, but requires means to uniformly distribute the inlet gas across the inlet side of the packed bed, and means to uniformly withdrawal the fuel gas from the packed bed to ensure uniformity of flow. This is usually done with large distribution plates and layers of large pellet beds that also support the adsorbent bed, and result in increased pressure drop across the reactor vessel.
- The packed bed reactor must be designed to maximize the sorbent bed capacity, so that the number of vessels and the frequency of switching is minimized, and to maintain acceptable vessel pressure drop. This is most economically accomplished in shop-fabricated vessels, limiting the maximum vessel outer diameter to about 4.9 m (16-ft).
- The packed bed reactor is prone to plugging if the inlet fuel gas contains any amount of entrained particulate, and even with almost particulate-free fuel gas will result in gradual increasing pressure drop due to corrosion products, chemical deposition, and settling of the packed bed.
- The packed bed reactor is a source of particle emission into the fuel gas. Even with low fuel gas velocities through the packed bed, the bed particles are subject to high levels of crushing forces and locally high gas velocities near the gas distribution plates, as well as particle chemical decrepitation within the bed, and such particle emissions cannot be tolerated in the IGCC application.

The proposed filter-reactor acts like a semi-continuous packed bed reactor, with each filter cake section being a continuously fed packed bed reactor that is periodically removed from service for an instant. The filter-reactor has no tendency for plugging and maintains particle-free fuel gas conditions. This evaluation indicates the filter-reactor has a high potential to provide high levels of contaminant removal performance.

Specifically, the filter-reactor evaluation has indicated the following:

- Significant HCl removal is expected in the entrained zone of the Halide Filter-Reactor, and the outlet fuel gas HCl content should be easily maintained below the required 5 ppmv level.
- The face velocity of the filter-reactor does not significantly influence the performance of contaminant removal in the filter-reactor, the recovery time for a plenum increasing only little as the face velocity is increased. Increased face velocity does increase the pulse cleaning frequency significantly, though.
- Only little sulfur removal is expected in the entrained zone of the Sulfur Polishing Filter-Reactor. The one-hour time averaged sulfur content of the outlet fuel gas is expected to be much below the required level of 2 ppmv, but the H<sub>2</sub>S level may jump above the required sulfur content momentarily during pulse cleaning events.
- No mercury removal is expected in the entrained zone of the Mercury Removal Filter-Reactor. Because the sorbent-to-mercury ratio can be maintained very high by recirculation, the level of mercury should always be maintained below the required level of control.
- The filter-reactor can perform multiple processing tasks that can improve IGCC performance and economics.
- The filter-reactor contaminant removal behavior, and the influence of the sorbent reaction products on the barrier filter performance need to be determined in PDU testing of prototype equipment.

# 5.5 PERFORMANCE ESTIMATES FOR IGCC WITH NOVEL GAS CLEANING

The breakdown of power generation and power use in the IGCC power plant with Filter-Reactor Novel Gas Cleaning technology is shown in Table 5.34. Compared are Novel Gas Cleaning meeting Current Standards and Future Standards. Overall, the lower sulfur emissions of the Future Standards case result in almost no change in plant heat rate, power plant thermal efficiency, or net plant power. The impact of the Future Standards is small because the only major change in the process is the insertion of the stage for sulfur polishing. Note that the Sulfur Recovery Section net power use is nearly zero, with the acid gas expander's generation balancing the power losses in the system.

Section	Current Standards Power (MWe)	Future Standards Power (MWe)
Power Island Generation		
Turbine Air Compressor	-171.60	-171.57
Gas turbine	374.89	374.83
Steam turbine	149.22	149.22
Generator	-7.1	-7.0
BOP	-5.2	-5.2
Total power	340.2	340.2
Air Separation Consumption		
ASU Air compressor	0	0
Oxygen compressor	4.129	4.129
Nitrogen compressor	8.98	8.98
ASU	20.0	20.0
Total power use	33.1	33.1
Gasification Consumption		
Fans & blowers	0.52	0.51
Pumps	0.094	0.094
Coal handling and preparation	1.5	1.5
Ash handling	1.3	1.3
Total power use	3.4	3.4
Desulfurization Consumption		
Refrigeration	0.0	0.0
Compressors	2.6	2.6
Pumps	0	0
Total power use	2.6	2.6
Sulfur Recovery Consumption		
Expander	-2.81	-2.81
Compressors	2.64	2.64
Pumps	0.00	0.00
Total power use	-0.17	-0.17
Total Plant		
Net plant power generation, MW	302.5	302.5
Plant net heat rate (HHV), kJ/kWh (Btu/kWh)	9029 (8558)	9029 (8558)
Plant net efficiency, % (LHV)	43.03	43.03
Plant net efficiency, % (HHV)	39.87	39.87

Table 5.34 - IGCC Power Pla	nt Thermal Performance	e with Novel Gas Clo	eaning Technology

Table 5.35 lists several quantities related to the use of resources and emissions in the IGCC power plant with Novel Gas Cleaning technology. The power plants in both cases use lower quantities of LP-steam in the gas cleaning process steps than does conventional gas cleaning technology. Cooling water use in the processing steps is also lower. Only small quantities of process condensate are generated in both cases, primarily in the Sulfur Recovery Section, and this is recycled without treatment. A moderate level of fresh process water is required in the gas cleaning processes, and both cases result in no discharge of process water. Both cases require small quantities of boiler-quality makeup water for nitrogen humidification.

The total sulfur emissions are expressed in three different bases (percent removal, mass per unit coal fuel input, and mass per MWe net power generated), and are significantly lower for the Future Standard case -- comparable to the sulfur emissions from a natural gas-fired combined-cycle power plant. Solid waste rates from the IGCC power plants are identical for both cases.

	Current	Future
	Standards	Standards
Auxiliaries		
Net IP steam use, MJ/hr (10 <sup>6</sup> Btu/hr)	40,305 (38.2)	40,305 (38.1)
Net LP steam use, MJ/hr (10 <sup>6</sup> Btu/hr)	-25,744(-24.4)	-25,744 (-24.4)
Total cooling water use, MJ/hr (10 <sup>6</sup> Btu/hr)	10,899 (103.3)	10,899 (103.2)
Total condensate generated, kg/hr (lb/hr)	2,463 (5,430)	2,465 (5,435)
Total process water used, kg/hr (lb/hr)	42,305 (93,266)	42,306 (93,268)
Net process water makeup, kg/hr (lb/hr)	39,842 (87,836)	39,841 (87,833)
Total boiler water makeup, kg/hr (lb/hr)	33,566 (74,000)	33,566 (74,000)
Emissions		
Sulfur total removal efficiency, %	99.57	99.983
Sulfur total emission (HHV),	11.38 (0.02648)	0.464 (0.00108)
mg/MJ (lb SO <sub>2</sub> / $10^6$ Btu)		
Sulfur total emission, kg/MW (lb SO <sub>2</sub> / MW)	0.0239 (0.2295)	0.00418 (0.00921)
NOx total emission (HHV),	15.22 (0.0354)	5.073 (0.0118)
mg/MJ (lb NO $_2$ / $10^6$ Btu)		
Particulate emission (HHV),	0.0086 (2E-05)	0.0086 (2E-05)
mg/MJ (lb/ $10^6$ Btu)		
Hg emission, mg/MJ (lb/TBtu) (HHV)	0.218 (0.507)	0.1088 (0.253)
Consumables, kg/hr (lb/hr)		
Chemicals	0	0
Sorbents	2,070 (4,564)	2,087 (4,600)
Catalyst	0.9 (2)	0.9 (2)
Solid waste, kg/hr (lb/hr)		
Slag product (25 wt% water)	13,673 (30,144)	13,673 (30,144)
Slag & HCl sorbent waste	1,706 (3,760)	1,706 (3,760)
Waste salts (25 wt% water)	0	0
Sorbent wastes	53 (117)	71 (156)
Total	15,432 (34,021)	15,450 (34,060)

Table 5.35 - IGCC Power Plant Resource Use and Emissions with Novel Gas Cleaning

## 5.6 COST ESTIMATES FOR IGCC WITH NOVEL GAS CLEANING

The costs for the Novel Gas Cleaning process and for the entire IGCC power plant using the Novel Gas Cleaning technology are displayed in Tables 5.36 through 5.38. Table 5.36 lists the purchased equipment and installed equipment costs, broken down to each section of the Novel Gas Cleaning process, for the Current and Future Standards cases. The total installed cost of the Future Standards case is about 19% greater than that of the Current Standards case. The Mercury Removal Section used in the cost results presented is the "once-through, powdered mercury sorbent" system.

Plant Section	Current Standards	Future Standards	
	Cost, k\$	Cost, k\$	
Raw Gas Convective Cooling			
purchased equipment	239	239	
installed equipment	478	478	
Desulfurization			
purchased equipment	5,959	8,737	
installed equipment	9,676	13,734	
Sulfur Recovery			
purchased equipment	4,705	4,548	
installed equipment	8,747	8,590	
Mercury Removal			
purchased equipment	1,430	1,442	
installed equipment	2,013	2,038	
Total Gas Cleaning			
purchased equipment	12,333	14,966	
installed equipment	20,914	24,840	
purchased equipment, \$/kW	41	49	
installed equipment, \$/kW	69	82	
Total Plant			
TCR, k\$	428,179	434,087	
TCR, \$/kW	1415	1435	

 Table 5.36 - Novel Gas Cleaning Technology Equipment Cost Breakdown

Cost estimates have been made for the Mercury Removal Section once-through and regenerative sorbent processes described, and are listed in Table 5.37 for the Future Standards case. Also shown are the equipment costs for comparable processes using pelletized mercury removal sorbents in packed bed reactors. The packed bed processes are significantly more expensive than the use of powdered sorbents in a filter-reactor process configuration.

	Future Standards
	Cost, k\$
Once-through, Powdered Sorbent (filter-reactor)	2,038
Regenerative, Powdered Sorbent (filter-reactor)	2,671
Once-through, Pelletized Sorbent (packed bed reactor)	3,473
Regenerative, Pelletized Sorbent (packed bed reactor)	7,105

The IGCC power plant Total Capital Requirement is computed in Table 5.36 for the Current and Future Standards cases. The Total Capital Requirement is about 1.5% greater in the Future Standards case than in the Current Standards case. The cost-of- electricity (COE) is estimated in Table 5.38 for the two cases, using a "first year of operation" basis. The COE is about 1.0% higher in the Future Standards case than in the Current Standards case.

	Current Stor dondo	Future Stor doudo
	Standards	Standards
	(Cents/kWh)	(Cents/kWh)
Fixed O&M	0.48	0.48
Variable O&M	0.27	0.27
Consumables	0.34	0.38
water	0.04	0.04
chemicals, sorbents, catalysts	0.16	0.20
waste Disposal	0.11	0.11
chemicals for BFW, CW and	0.03	0.03
waste treatment		
Sulfur by-product credit	0.06	0.06
Fuel	1.59	1.59
Capital charges	3.73	3.78
Total	6.34	6.40

Table 5.38 – Cost-of-Electricity for IGCC with Novel Gas Cleaning

# 5.7 ASSESSMENT OF IGCC WITH NOVEL GAS CLEANING

All four of the IGCC case simulations have been with respect to the quality of the simulations:

- validity of process flow diagrams,
- major component pressure drop estimates,
- pulse gas and transport gas rate estimates,
- once-though and regenerative mercury removal scheme assumptions,
- overall material & energy balance convergences,
- gas turbine scaling,
- plant steam balances and steam turbine cycle conditions,
- plant performance calculations.

The results are valid as a means for "relative" comparison to indicate potential merits and issues. A consistent design basis and emissions control approach has been applied so that overall power plant performance and cost comparisons are meaningful and representative of actual expectations on a relative basis. The evaluation shows that the Filter-Reactor Novel Gas Cleaning technology provides reduction in IGCC power plant complexity, increase in power plant generating capacity and thermal efficiency, and reduced investment and cost-of-electricity over IGCC power generation using conventional dry-gas cleaning technology. Expected are power plant availability advantages for the IGCC power plant using Filter-Reactor Novel Gas Cleaning technology, with the technology providing greater gas turbine protection than the conventional gas cleaning technology.

## 5.7.1 Performance Potential

The IGCC overall performance results are tabulated in Table 5.39. The results indicate that the Novel Gas Cleaning schemes have the potential for significant improvements in IGCC power plant generating capacity and heat rate. Novel Gas Cleaning yields 6% greater generating capacity and 2.3 percentage-points greater efficiency under the Current Standard case, and more than 9% generating capacity and 3.6 percentage-points higher efficiency in the Future Standards case. Note that Novel Gas Cleaning performance is almost entirely insensitive to the gas cleaning standards, with little difference between the Current and Future Standards cases. Solid waste from IGCC with Novel Gas Cleaning is about 8% greater than from IGCC with conventional gas cleaning.

	Conventional	Conventional	NGC	NGC
	Cold Gas	Cold gas		
	Cleaning	Cleaning		
Standards	Current	Future	Current	Future
Net Power, MW	285	276	303	303
Net Eff, % (HHV)	% (HHV) 37.6		39.9	39.9
Solid Waste, kg/hr	14,288	14,288	15,432	15,450

 Table 5.39 - Overall IGCC Performance Comparison

The comparison of power plant performance between a conventional, natural gas combined-cycle and the four IGCC power plants are shown in Tables 5.40 and 5.41. Table 5.40 focuses on water resource use, power generating capacity, and thermal efficiency factors for each power plant. Table 5.41 focuses on power plant environmental factors.

Table 5.40 shows that the Novel Gas Cleaning technology provides the potential for improvement in IGCC power plant water resource use and thermal performance over IGCC using conventional gas

cleaning technology. The Novel Gas Cleaning technology uses less than half the cooling water rate used by the conventional gas cleaning technology. Total water consumption is about the same for both technologies. Novel Gas Cleaning technology consumes more process makeup water than the conventional gas cleaning technology, primarily for coal slurry preparation, but it uses much less boilerquality makeup water, a more expensive water source.

The detailed power consumption breakdowns listed in this report indicate that every section of the fuel gas cleaning process, except for the sulfur recovery process, shows significant advantage for the Novel Gas Cleaning technology. Conventional fuel gas cleaning technology applies a large number of fuel gas cooling and reheating operations, conducting total condensation of the fuel gas water vapor, followed by re-humidification, and this results in significant losses in overall power plant thermal efficiency that do not occur when using the Novel Gas Cleaning technology. When meeting the fuel gas cleaning requirements of the Current Standards case, the IGCC power plant with Novel Gas Cleaning technology produces 17.3 MWe greater power output than the plant using conventional gas cleaning, a 6.1% increase in power. Similarly, the Novel Gas Cleaning technology results in a 2.3 percentage-point increase in power plant efficiency (HHV) over the IGCC power plant using conventional gas cleaning technology.

	Natural Gas	Conventional	Novel Gas	Conventional	Novel Gas
	Combined-	Cleaning	Cleaning	Cleaning	Cleaning
	Cycle	Current Standards	Current Standards	Future Standards	Future Standards
Cooling water use,	0	252,169	108,675	199,414	108,675
MJ/hr					
Process water	0	-274	42,305	0	42,306
Makeup, kg/hr					
Boiler feed water	0	69,263	33,566	69,646	33,566
Makeup, kg/hr					
Total water	0	68,989	75,872	69,646	75,873
consumption, kg/hr					
Net power generated,	272.3	285.2	302.5	275.5	302.5
MW					
Plant net heat rate,	6581	8876	8367	9187	8366
kJ/kWh (LHV)					
Plant net efficiency,	54.7	40.6	43.0	39.2	43.0
% (LHV)					
Plant net efficiency,		37.6	39.9	36.3	39.9
% (HHV)					

 Table 5.40 - Natural Gas Combined-Cycle and IGCC Fuel Gas Cleaning Plant Comparisons

Even greater advantages result when meeting the gas cleaning requirements of the Future Standards case. Here, the IGCC power plant with Novel Gas Cleaning technology produces 27.0 MWe greater power output than the plant using conventional gas cleaning, a 9.8% increase in power. Similarly, the Novel Gas Cleaning technology results in a 3.6%-point increase in power plant efficiency (HHV) over the IGCC power plant using conventional gas cleaning.

The evaluation basis calling for the Novel Gas Cleaning Process to produce the final sulfur product in the form of elemental sulfur makes the sulfur recovery process the largest contributor to thermal performance reduction in the Novel Gas Cleaning Process. If other products were acceptable

sulfur forms, such as sulfuric acid, the process would show even greater thermal performance advantages over the conventional gas cleaning technology.

The IGCC power plants (all four cases) generate 3 to 30 MWe more electric power than the natural gas combined-cycle power plant using the same gas turbine and steam turbine conditions, but at 12 to 15 percentage-points lower thermal efficiency. The natural gas-fired power plant requires no gas cleaning and thus there are no cooling water, process water, of boiler feed water makeup flow associated the gas cleaning for the natural gas combined-cycle power plant.

Table 5.41 lists the clean fuel gas and stack gas compositions estimated for the IGCC power plant cases, and compares them to the conventional, natural gas combined-cycle. With a typical natural gas sulfur content of 7 ppmv, the IGCC stack emissions of sulfur oxides for the Future Standards case approach the emissions of sulfur oxides from the natural gas combined-cycle power plant. It should be noted that natural gas is processed before use in the combined-cycle power plant to remove sulfur species, water vapor, carbon dioxide, and mercury, and this processing results in various emissions of carbon dioxide, sulfur species that are not reflected in this comparison.

The conventional gas cleaning technology has established capability to achieve the levels of sulfur control required in the evaluation. The Filter-Reactor Novel Gas Cleaning technology is estimated as having this capability based on the development work completed. Where these gas cleaning technologies differ is that the conventional gas cleaning technology will reduce the fuel gas halide, ammonia, and HCN contents to much lower levels in the fuel gas than will the Novel Gas Cleaning technology. The Novel Gas Cleaning technology can, though, reduce the fuel gas halide, ammonia and HCN contents sufficiently to satisfy the requirements for IGCC applications. The Novel Gas Cleaning technology uses partial-decomposition of ammonia in the process, and low-NOx, diffusion flame, fuel gas combustors (staged, rich-quench-lean; or catalytic) to achieve low NOx emissions from the IGCC power plant. The higher-temperature operations of the Novel Gas Cleaning technology may provide advantages, with less potential for the formation of metal carbonyls, and its final stage Mercury Removal Filter-Reactor may provide additional protection against corrosion-based particulate damage to the gas turbine not provided by conventional gas cleaning technology. The conventional gas cleaning technology, with operation at conditions of high corrosion potential, is inherently more prone to availability losses than the Novel Gas Cleaning technology.

All of the IGCC power plants produce comparable solid waste streams, with the total mass of waste for the Novel Gas Cleaning technology being about 8% greater than with the conventional gas cleaning. The nature of the waste differs slightly for the two technologies. The slag waste streams are very similar in flow rate and composition for the four IGCC power plants. The conventional gas cleaning technology produces a wet stream of halide salts that contain numerous traces of contaminants, as well as a small, hazardous mercury sorbent waste. The Novel Gas Cleaning technology produces dry, non-hazardous sorbent waste, some of which can be incorporated into the plant slag waste. The hazardous nature of the mercury sorbent waste from the Novel Gas Cleaning Process is uncertain at this time.

## 5.7.2 Cost Potential

While the equipment costs are estimated to be only slightly lower for the Novel Gas Cleaning processes than for the conventional processes, the improved power plant capacity results in the potential for significant reductions in the plant cost-of-electricity.

The IGCC power plant investment and cost-of-electricity (COE) is compared for the four plants in Table 5.42. The plant meeting Current Standards has 6% lower investment when using the Novel Gas Cleaning technology. For the Future Standards case, the capital investment is almost 13% lower for the Novel Gas Cleaning technology.

The COE is reduced by about 3% in the Current Standards case by using the Novel Gas Cleaning technology. Almost 8% reduction is found for the Future Standards case. It is also seen that IGCC using

	Natural Gas	Current Standards		Future Standards	
	Combined-Cycle	Conventional	Novel	Conventional	Novel
Clean Fuel Gas		Cleaning	Cleaning	Cleaning	Cleaning
(before N <sub>2</sub> dilution)					
$H_2$ (vol%)	0	25.2	22.9	25.2	22.9
CO (vol%)	0	43.7	36.7	43.7	36.7
CH <sub>4</sub> (vol%)	98	0.3	0.3	0.3	0.3
CO <sub>2</sub> (vol%)	0	9.3	10.3	9.3	10.3
$H_2O$ (vol%)	0	19.7	19.5	19.7	19.5
N <sub>2</sub> (vol%)	2	1.7	10.1	1.7	10.1
Ar (vol%)	0	0.14	0.19	0.14	0.2
Total sulfur (ppmv)	7	102.6	42.9	2.1	1.7
Halides (ppmv)	0	0.1	3.5	0.1	3.5
Ammonia (ppmv)	0	0.4	158	0	158
HCN (ppmv)	0	0	19	0	19
Hg (ppbv)	0	0.29	0.26	0.146	0.129
Stack Gas					
CO <sub>2</sub> (vol%)	3.8	9.2	9.0	9.5	9.0
$H_2O$ (vol%)	8.4	11.7	12.0	11.7	12.0
N <sub>2</sub> (vol%)	74.1	70.2	70.0	70.2	70.0
O <sub>2</sub> (vol%)	12.2	8.1	8.2	8.0	8.2
HCl (ppmv)	0	0	0.7	0	0.7
SO <sub>2</sub> (ppmv)	0.26	17.7	8.2	0.36	0.33
NO (ppmv)	<15	<15	<15	<5	<5
Hg (ppbv)	0	0.05	0.05	0.026	0.025
Sulfur Removal					
Total removal efficiency (%)	0	99.08	99.57	99.981	99.983
Total emission	0.494	24.506	11.522	0.503	0.464
(mg SO <sub>2</sub> / MJ)					
Solid Waste					
Slag and flyash	0	14,015	13,673	14,015	13,673
(wet, kg/hr)	0	295	0	295	0
Waste salts (wet, kg/hr)	U	285	0	285	0
Sorbent wastes	0	1.4	1,713	1.4	1,708
(kg/hr)	U	1.4	1,/15	1.4	1,700
Total (kg/hr)	0	14,301	15,386	14,301	15,381

 Table 5.41 - Natural Gas Combined-Cycle and IGCC Fuel Gas Cleaning Emission Comparisons

the Novel Gas Cleaning technology could achieve the Future Standards at about the same investment and COE as the conventional IGCC power plant that achieves only Current Standards.

Gas Cleaning Technology	Conventional Cleaning	Novel Cleaning	Conventional Cleaning	Novel Cleaning
Gas Cleaning Standards	Current	Current	Future	Future
Generation capacity, MWe	285.2	302.5	275.5	302.5
Plant Heat Rate, kJ/kWh (HHV)	9574	9022	9917	9022
Total Capital Requirement, \$/kW	1500	1415	1614	1435
Total COE, cents/kWh (constant \$)	6.56	6.34	6.93	6.40

Table 5.42 - Total IGCC Power Plant Investment and COE

# 6. METHANOL SYNTHESIS WITH CONVENTIONAL GAS CLEANING

The focus of the evaluation now turns to Novel Gas Cleaning for coal-based chemical synthesis in Sections 6 and 7. The chemical synthesis application evaluated is a methanol synthesis plant combined with electric power production. The Liquid Phase Methanol synthesis process is the methanol synthesis technology applied in the evaluation. The plant is generally similar to the IGCC power plant evaluated in Sections 4 and 5, except that 1) it operates at a higher pressure to generate a clean fuel gas stream and a clean synthesis gas stream that can be utilized for methanol synthesis without syngas compression, 2) it achieves more stringent gas cleaning standards, and 3) the power island utilizes the fuel energy in the clean fuel gas steam and in the purge gas from the methanol synthesis portion of the plant. The nominal capacity of the plant is 300 MWe electric power, and 318 tonnes (350 tons) of methanol (fuel grade) per day. The plant chemical synthesis process requires stringent cleaning of sulfur species, halide species, particulate, ammonia, hydrogen cyanide, mercury, and metal carbonyls.

The questions addressed by the evaluation are:

- How is the Novel Gas Cleaning technology best configured to meet the chemical synthesis gas cleaning requirements?
- How does the resulting Novel Gas Cleaning process compare to conventional synthesis gas cleaning technology with respect to performance and cost potential?
- What are the major development issues for the Novel Gas Cleaning technology for chemical synthesis applications?

Two plant configurations have been evaluated in addressing these issues:

- a plant using conventional, low-temperature gas cleaning technology, in Section 6,
- a plant using the Siemens Novel Gas Cleaning technology to meet the methanol synthesis requirements, in Section 7 of the report.

# 6.1 METHANOL SYNTHESIS PLANT WITH CONVENTIONAL GAS CLEANING

The overall methanol synthesis process flow diagram with conventional gas cleaning is illustrated in Figure 6.1. The plant consists of six major sections: Gasification, Low-Temperature Cooling, Acid Gas Removal (AGR), Sulfur Recovery, Methanol Synthesis, and Power. It is similar to the conventional IGCC power plant overall process diagram, except that

- a greater coal feed rate is consumed to accommodate the fuel needs for both power generation and methanol synthesis,
- cleaned gas is split into a "syngas" stream for methanol synthesis and a "fuel gas" for power generation, and
- the gasifier is operated at a much higher pressure to generate syngas that can meet the high-pressure needs of the methanol synthesis reactor without syngas compression.

The methanol synthesis gas cleaning requirements are those outlined in Section 3.2 of the report. The raw gas is desulfurized using Rectisol desulfurization technology to a lower sulfur content than in the IGCC Future Standards case (Report Section 4). The stream of syngas split from the cleaned gas is further cleaned of sulfur and metal carbonyls in fixed beds of sorbents before the syngas is conditioned and reacted for methanol synthesis. The methanol synthesis purge gas and the cleaned fuel gas streams are both expanded and combined in the Power Section before being fed to the Power Island gas turbine combustors. The recycling of process streams and the interchanging of heat between the major process sections are numerous and require complex integration.

# **GASIFICATION SECTION**

The Gasification Section primary function is to convert coal into a raw gas cooled to a suitable temperature for treatment in the subsequent Low-Temperature Cooling Section. Figure 6.2 shows the

process flow diagram for the Gasification Section. Table 6.1 lists characteristics of the major stream in the Gasification Section. The process diagram is identical to the configuration used in the conventional IGCC power plant evaluation.

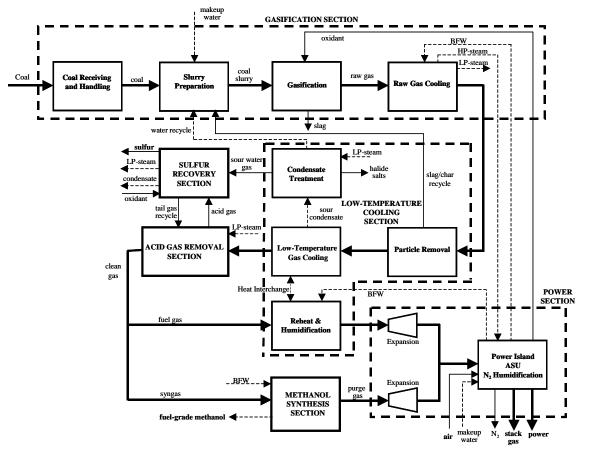


Figure 6.1 – Conventional Methanol Synthesis Overall Process Scheme

Oxidant from the Power Section's air separation unit has a flow of about 108,567 kg/hr (239,345 lb/hr). The raw gas flow rate at the exit of the Convective Cooler (Item 7) has a heat content of 634 MW(t) (2,165 x 10<sup>6</sup> Btu/hr) (LHV) and a heating value of about 7.82 MJ/Nm<sup>3</sup> (199 Btu/scf), and includes 11,612 kg/hr (25,600 lb/hr) of recycled, clean fuel gas used as soot blower gas in the raw gas heat exchangers. The raw gas exits the Gasifier (Item 3) at a temperature of about 1467°C (2582°F) and a pressure of 8791 kPa (1275 psia), and is cooled to about 282°C (540°F) at the exit of the Convective Cooler (Item 7), with the estimated composition in Table 6.2.

The estimated distribution of contaminants in the raw gas issued from the gasifier is based on the empirical assumptions listed in Table 6.3. Significant sulfur content is assumed to remain in the gasifier slag, and the hot gas from the gasifier is assumed to be at equilibrium with respect to sulfur species. All of the coal's chlorine content is assumed converted to HCl in the raw gas, and other halides have been neglected in the evaluation. All of the coal's mercury is assumed to be issued in the raw gas as elemental mercury. Empirical conversions are assumed for ammonia and hydrogen cyanide. Metal carbonyls may be formed in the raw gas only at temperatures less than 900°F though gas-solid reactions with the ash metal constituents, or with the materials of construction, and the level of formation is highly uncertain. The hot gas composition is assumed to be frozen at this level once it has passed through the raw gas cooler heat exchangers.



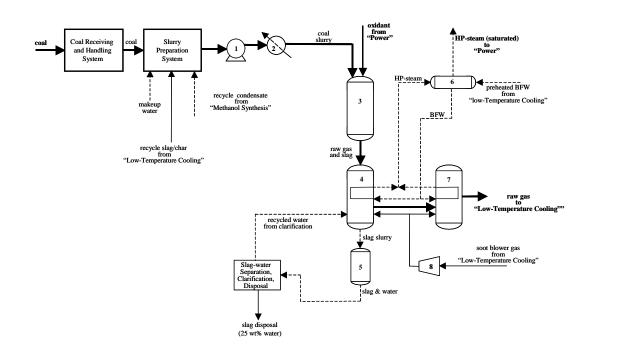


Figure 6.2 – Gasification Section - Conventional Methanol Synthesis

Table 6.1 – Stream	<b>Characteristics for</b>	<b>Gasification Section fo</b>	r Current Standards

Stream name	Coal feed	Total slurry	Gasifier	Raw fuel	Slag	HP-steam
		water feed	oxidant	gas		
Molar flow, kmole/hr	10,255	2,826	3,352	12,975	261	20,898
Mass flow, kg/hr	115,058	50,940	106,814	272,191	12,235	376,482
Volumetric flow, m <sup>3</sup> /hr	174	77	1,142	7,654	20	6,941
Temperature, °C	26	47	115	282	642	318
Pressure, kPa	101	110	9534	7922	7922	10,583
Enthalpy MJ/hr	-1.46E+05	-8.03E+05	7,714	-1.64E+06	-49,025	-4.99E+06
Molecular wt	11.2	18.0	31.9	21.0	46.9	18.0

	Major constituents (vol%)
H <sub>2</sub>	25.39
CH <sub>4</sub>	0.30
СО	41.03
$CO_2$	9.86
H <sub>2</sub> O	20.63
N <sub>2</sub>	1.58
Ar	0.14
Total	98.93
	Major contaminants (ppmv)
$H_2S$	9,020
COS	641
$CS_2$	0.6
S <sub>X</sub>	2
$SO_2$	1
NH <sub>3</sub>	670
HCN	19
HC1	435
Hg (ppbv)	3

 Table 6.2 - Raw Fuel Gas Composition – Conventional Methanol Synthesis

 Table 6.3 – Distribution of Contaminants in Gasification Section Raw Gas

Contaminant	Generation	Contaminant forms
	(% of coal constituent)	
Sulfur species	90% (10% retained with slag)	$H_2S$ , COS, CS <sub>2</sub> , SO <sub>2</sub> based on
		equilibrium at exit temperature
Halides	100% of coal Cl content	HCl (other halides neglected)
Ammonia	25 % of coal nitrogen	NH <sub>3</sub>
Hydrogen cyanide	0.5 % of coal nitrogen	HCN
Mercury	100% of coal mercury content	Hg <sup>°</sup> only
Metal carbonyls	0 (generated <900°F only)	Fe(CO) <sub>5</sub> , Ni(CO) <sub>4</sub>

## **Low-Temperature Cooling Section**

Figure 6.3 represents the process flow diagram for the Low-Temperature Cooling Section. Table 6.4 list characteristics of the major process streams in this section. This complex section cools the raw gas to the temperature required by the Desulfurization Section, and it functions to remove significant halides, ammonia, hydrogen cyanide, and mercury. The major equipment components are described below.

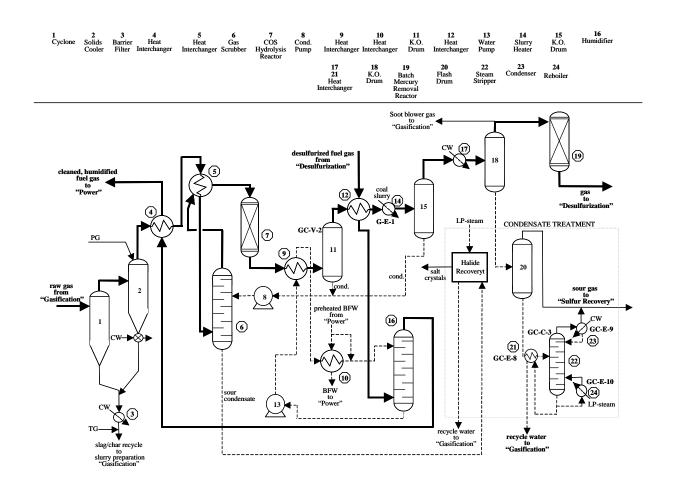


Figure 6.3 – Low Temperature Cooling Section - Conventional Methanol Synthesis

Stream name	Cooled	Slag/char	Fuel gas to	Desulfurized	Reheated fuel gas
	raw gas	recycle	Desulfurization	fuel gas	to Power Island
Molar flow, kmole/hr	13,025	50	8,536	9,759	11,397
Mass flow, kg/hr	275,173	2,982	191,746	212,044	241,942
Volumetric flow, m <sup>3</sup> /hr	7,211	2.0	9,032	3,525	8.965
Temperature, °C	282	282	38	38	360
Pressure, kPa	8,419	7,922	2,441	7,191	6,847
Enthalpy, MJ/hr	-1.66E+06	-14,446	-9.66E+05	-1.044E+06	-1.26E+06
Molecular wt	21.1	60.0	22.5	21.7	21.2

Table 6.4 – Stream Characteristics for Low-Temperature Cooling Section

<u>Barrier filter</u>: A ceramic, or metal, candle barrier filter (Item 2) is placed to follow a conventional cyclone (Item 1) and operates at about 282°C (540°F) to removal particulate (solidified slag particles and char) from the raw gas to a level of < 0.1 ppmv as the first step in the cleaning process.

<u>Fuel gas coolers</u>: A process heat interchanger (Item 4) is now used to cool the gas to about  $241^{\circ}$ C (467°F), while reheating the clean fuel gas stream to about  $271^{\circ}$ C (520°F) before it goes to the Power

Section. This cooled gas then passes through a second heat interchanger (Item 5) that cools it to about 219°C (426°F).

<u>Gas condensate scrubber</u>: The gas is next scrubbed in a column (Item 6) with collected process condensate to remove halides to a very low level, and results in the further cooling of the gas to about  $203^{\circ}$ C (397°F).

<u>Gas reheater</u>: The gas is now reheated in a gas heat interchanger (Item 5) to about 225°C (437°F) in preparation for COS hydrolysis. This reheat is dictated by a need to heat the saturated gas to minimize the possibility of condensate formation in the hydrolysis reactor.

<u>COS hydrolysis</u>: The reheated gas is treated in a catalytic reactor (Item 7) to hydrolyze its COS content to  $H_2S$ , allowing more efficient sulfur removal to be performed downstream. This also results in substantial HCN hydrolysis to NH<sub>3</sub>.

<u>Gas cooling and ammonia removal</u>: Next, a series of process heat interchangers and water-cooled heat exchangers (Items 9, 12, 14, and 17 and their associated knock-out drums) are used to cool the gas to about 38°C (100°F), simultaneously removing most of the gas water content and collecting process condensate that contains most of the gas ammonia. A Stream of released gases from condensate processing (sour-gas water), at 388 kg/hr (856 lb/h)r, is sent to the Sulfur Recovery Section of the plant. The composition of this sour-water gas is listed in Table 6.5.

	Constituents (vol%)
$H_2$	1.62
$CH_4$	0.03
CO	2.85
$CO_2$	20.87
H <sub>2</sub> O	50.30
$N_2$	0.08
$H_2S$	9.12
COS	0.10
NH <sub>3</sub>	15.02
HCN	0.00
Total	99.99

# Table 6.5 - Sour-water Gas Composition – Conventional Methanol Synthesis

<u>Mercury removal</u>: The gas is now treated in a fixed bed reactor (Item 19) containing sulfur-impregnated, activated carbon, and sized to remove 95% of the gas mercury (Smith, 2000).

The fates of the gas contaminants in the raw gas entering this section as estimated in the process simulation are listed in Table 6.6. The halides, ammonia, and hydrogen cyanide contaminants are very effectively removed from the raw gas, and are recovered from the condensate streams for final processing. The halides are converted to ammonia salts for disposal, and ammonia is sent to the Sulfur Recovery Section to be decomposed in the high temperature Claus furnace. Considerable LP-steam is utilized in the condensate treatment process. The composition of the gas sent to the Desulfurization Section of the plant is listed in Table 6.7.

Contaminant	Removal	Contaminant conversions
	(% of inlet to Section)	
sulfur species	6.1	Hydrolysis
		Partial condensate absorption
halides	100	Condensate absorption
		Salt crystallization
ammonia	99.05	Hydrolysis
		Condensate absorption
		Salt crystallization
Hydrogen cyanide	100	Hydrolysis
mercury	98	Sorbent fixation

	Major constituents (vol%)
H <sub>2</sub>	31.99
CH <sub>4</sub>	0.38
СО	51.70
CO <sub>2</sub>	12.47
H <sub>2</sub> O	0.09
N <sub>2</sub>	1.99
Ar	0.17
Total	98.79
	Major contaminants (ppmv)
$H_2S$	12,065
COS	0.0
NH <sub>3</sub>	8.5
HCN	0.0
Hg (ppbv)	0.1

<u>Humidification</u>: The clean and dry gas from the desulfurizer is split into two streams, one being "fuel gas", the other "syngas". The clean and dry fuel gas from the desulfurizer is reheated to about  $142^{\circ}C$  (288°F), interchanging heat (Item 12) with the previous gas cooling-condensation process streams. This stream is humidified in a column (Item 16) of countercurrent warm water, the exit gas being at 185°C (365°F).

<u>Fuel gas reheat</u>: The cleaned and humidified fuel gas is now reheated to about 271°C (520°F) in a heat interchanger (Item 4). The clean fuel gas composition is listed in Table 6.8.

# Acid Gas Removal (AGR) Section

Figure 6.4 shows the process flow diagram for the AGR Section when meeting the methanol synthesis cleaning standards. Table 6.9 list characteristics of the major stream in this section. The Rectisol desulfurization technology is selected for the desulfurization step, with other commercial low-temperature desulfurization technologies unable to meet the stringent requirements of this evaluation. Rectisol claims that this desulfurization process will also remove HCN, NH<sub>3</sub>, CS<sub>2</sub>, iron and nickel carbonyls, VOCs, and mercury very effectively (Koss and Meyer, 2003). On the other hand, users of

Rectisol continue to perform mercury removal in a separate unit placed before the Rectisol process, and place adsorbers to capture metal carbonyls after the Rectisol process to ensure performance. If these contaminants are absorber in the Rectisol solvent, they will either accumulate within the solvent leading to operating problems, and/or be released from the stripper and need separate processing to engage their final disposition. Maintenance of the Rectisol columns is a critical path item in a conventional methanol synthesis plant due to the severe conditions in the columns (Trapp et al., 2004).

	Major constituents (vol%)
H <sub>2</sub>	27.18
CH <sub>4</sub>	0.32
СО	43.82
CO <sub>2</sub>	10.63
H <sub>2</sub> O	16.18
N <sub>2</sub>	1.72
Ar	0.15
Total	100.00
	Major contaminants (ppmv)
$H_2S$	0.22
COS	0.02
NH <sub>3</sub>	0.4
HCN	0.0
Hg (ppbv)	0.1

Table 6.8 - Cleaned and Humidified Fuel Gas Composition – Conventional Methanol Synthesis

The Rectisol process contacts fuel gas recuperatively-cooled to about -32°C (-26°F) (Item 1) with refrigerated methanol at about -71°C (-95°F) (Item 8) in a multi-staged, bubble-cap tray, counter-current absorption column (Item 3). A small portion of methanol is injected directly into the fuel gas stream before heat interchanger Item 1 to remove water from the fuel gas (Item 2) to eliminate the possibility of ice formation. The desulfurized fuel gas achieves a level of about 0.3 ppmv total sulfur content. The desulfurized, reheated fuel gas exits the process (Item 1) at about 13°C (56°F). Makeup methanol solvent at 133 kg/hr (293 lb/hr) is fed to the process to account for process losses, and a methanol decontamination step is included in the process. The process uses significant energy for refrigeration associated with heat interchanger Item 8 and condenser Item 12 to meet this level of sulfur removal. The lean solvent is flashed in a series of three flash to low pressure, 103 kPa (15 psia) and is circulated to a solvent stripper column (Item 11). The stripper generates a low-pressure, acid gas that is sent to a sulfur recovery process.

 Table 6.9 – Stream Characteristics for AGR Section

Stream name	Fuel gas from	Desulfurized	Acid Gas to
	LT-Cooling	fuel gas	Sulfur Recovery
Molar flow, kmole/hr	9,759	9,654	625
Mass flow, kg/hr	212,045	208,384	26,216
Volumetric flow, m <sup>3</sup> /hr	3,530	3,287	8,143
Temperature, °C	38	14	22
Pressure, kPa	7,191	6,978	186
Enthalpy, MJ/hr	-1.044E+06	-1.050E+06	-1.99E+05
Molecular wt	21.7	21.6	41.9

Contaminant	Removal	Contaminant conversions
	(% of inlet to Section)	
Sulfur species	99.9978	Methanol stripper
Halides	NA	
Ammonia	99.1	None
Hydrogen cyanide	NA	
Mercury	100	Accumulates in methanol

 Table 6.10 – Fate of Contaminants in the Conventional Desulfurization Section

Table 6.11 lists the composition of the desulfurized fuel gas. Table 6.12 lists the composition of the acid gas sent to the Sulfur Recovery Section.

	Major constituents (vol%)
H <sub>2</sub>	32.43
$CH_4$	0.39
СО	52.27
CO <sub>2</sub>	12.68
H <sub>2</sub> O	0.00
$N_2$	2.05
Ar	0.18
Total	100.00
	Major contaminants (ppmv)
$H_2S$	0.26
COS	0.03
NH <sub>3</sub>	0.4
HCN	0.0
Hg (ppbv)	0.1

 Table 6.11 - Desulfurized Fuel Gas Composition – Conventional Methanol Synthesis

 Table 6.12 - Acid Gas Composition - Conventional Methanol Synthesis

	Major constituents (vol%)
H <sub>2</sub>	0.00
CO	0.05
CO <sub>2</sub>	79.34
H <sub>2</sub> O	0.00
$H_2S$	19.89
COS	0.02
NH <sub>3</sub>	0.07
HCN	0.00
Methanol	0.63
Total	100.00

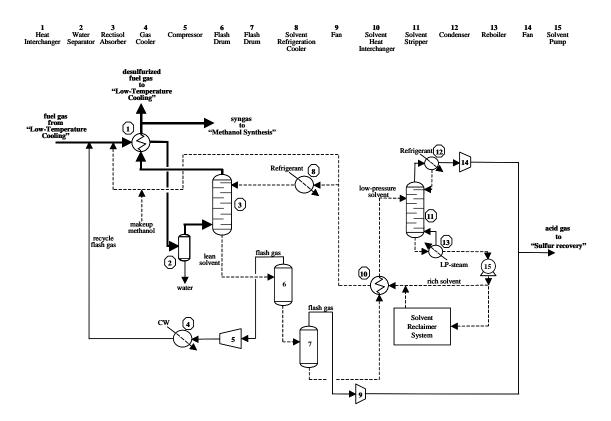


Figure 6.4 – AGR Section - Conventional Methanol Synthesis

## **Methanol Synthesis Section**

Figure 6.5 illustrates the Methanol Synthesis Section of the plant. Table 6.13 lists characteristics of the major streams in this section. Liquid Phase Methanol synthesis technology is applied. Syngas, at 13°C (56°F), is split from the AGR Section product stream, is passed through a metal carbonyl adsorption bed (Item 1), followed by a process stream heat interchanger (Item 2) that warms the syngas to 204°C (400°F), and a sulfur adsorption bed (Item 3) that polishes the syngas to meet methanol synthesis sulfur requirements. This syngas is then humidified to greater than 9 vol% water vapor in a column of circulating boiler feed water (Item 4). This circulating water is heated by internal heat transfer surface placed in the Liquid Methanol synthesis reactor (Item 7). The humidified syngas passes through the methanol synthesis reactor where about 41% of the syngas hydrogen is converted to methanol. In addition to heating circulating boiler feed water, LP-steam is generated in the methanol reactor fluid bed, controlling the exit gas temperature to about 249°C (480°F). A cyclone captures and recycles elutriated catalyst particles and oil to the bed. The methanol reactor product gas is then cooled to about 99°C (210°F) and oil is separated from the product gas in a separation vessel (Item 9). The product gas is further cooled to about 38°C (100°F) before passing through a knock-out vessel (Item 12) to separate the synthesis liquid and purge gas products. The purge gas from the knock-out vessel is reheated to about 87°C (189°F) before passing to the Power Section.

The fate of the contaminants within the Methanol Synthesis Section are listed in Table 6.14. The purge gas composition is presented in Table 6.15, and has a heating value of about 8.73 MJ/Nm<sup>3</sup> (222 Btu/scf). The synthesis product is flashes to about 117 kPa (17 psia) (Item 13) to separate some dissolved gases, at about 109 kg/hr (240 lb/hr) from the crude methanol product. The crude methanol is distilled (Item 14) to generate the fuel-grade methanol product. In total, 93.0 % of the methanol generated in the synthesis reactor is recovered in the final product.

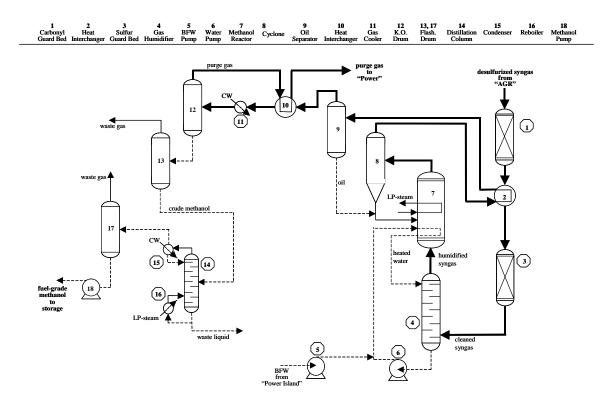


Figure 6.5 – Methanol Synthesis Section - Conventional Methanol Synthesis

Stream name	Desulfurized	Methanol	Purge gas to
	fuel gas feed	product	Power Section
Molar flow, kmole/hr	4.427	280	3,973
Mass flow, kg/hr	95,566	8970	94,220
Volumetric flow, m <sup>3</sup> /hr	1,504	12	1,855
Temperature, °C	13	38	87
Pressure, kPa	6,978	345	6,440
Enthalpy, MJ/hr	-4.82E+05	-67,731	-5.64E+05
Molecular wt	21.6	32.1	23.7

Table 6.13 – Stream Characteristics for AGR Section

 Table 6.14 – Fate of Contaminants in Methanol Synthesis Section

Contaminant	Removal	Contaminant
	(% of inlet to Section)	conversions
Metal carbonyls	90	Sorbent fixation
Sulfur species	90	Sorbent fixation
Halides	NA	
Ammonia	NA	
Hydrogen cyanide	NA	
Mercury	NA	

	Major constituents (vol%)	
H <sub>2</sub>	31.87	
CH <sub>4</sub>	0.43	
CO	39.76	
CO <sub>2</sub>	24.98	
H <sub>2</sub> O	0.01	
$N_2$	2.29	
Ar	0.20	
methanol	0.45	
Total	99.99	

Table 6.15 – Purge Gas Composition - Conventional Methanol Synthesis

The composition of the fuel-grade methanol is listed in Table 6.16. The composition is reported on a weight-percent basis and meets all specifications for fuel-grade methanol.

	Major constituents (wt%)
H <sub>2</sub> O	0.55
$CO_2$	0.04
Methanol	98.00
Methyl formate	0.18
Ethanol	1.23
Total	100.00

Table 6.16 – Methanol Composition with Conventional Cleaning

#### **Sulfur Recovery Section**

Figure 6.6 shows the process flow diagram for the Sulfur Recovery Section of the plant. It is almost identical in configuration to the Sulfur Recovery Section described for IGCC application with conventional gas cleaning, only with increased flow capacity of about 16%. The sour-water gas, containing significant ammonia and  $H_2S$ , and an appropriate portion of the acid gas are combined and are burned in the Claus furnace (Item 2) with a stream of oxygen, at 1,750 kg/hr (3,857 lb/hr) from the air separation unit. The sulfur recovery process recovers about 94.6% of the sulfur content of the acid gas. The collected sulfur streams are combined and the sulfur, at 4,096 kg/hr (9,031 lb/hr) is stored for treatment and marketing as a by-product.

The untreated tail gas, at 24,253 kg/hr (53,467 lb/hr), contains a considerable content and variety of sulfur species ( $H_2S$ ,  $SO_2$ ,  $S_x$ ), and the only way the power plant can achieve its overall 99.85% sulfur removal goal is for this tail gas to be recompressed and recycled to the desulfurizer. A catalytic hydrolyzer bed (Item 20), operating at about 316°C (600°F) is used to eliminate the oxidized sulfur species, and requires an additional oxygen feed stream of 363 kg/hr (800 lb/hr) to conduct partial oxidation to bring the gas to sufficient temperature.

The fates of the contaminants in the Sulfur Recovery Section are listed in Table 6.17. After cooling and compression and condensate removal, the tail gas recycled to the Gasifier has the composition reported in Table 6.18.

Contaminant	Removal	Contaminant conversions
	(% of inlet to Section)	
Sulfur species	94.66	Claus conversion to elemental
_		sulfur
		Hydrolysis to reduced forms
Halides	NA	
Ammonia	86.5	Furnace decomposition to N <sub>2</sub>
Hydrogen cyanide	NA	
Mercury	NA	

 Table 6.17 – Fate of Contaminants in Conventional Sulfur Recovery Section

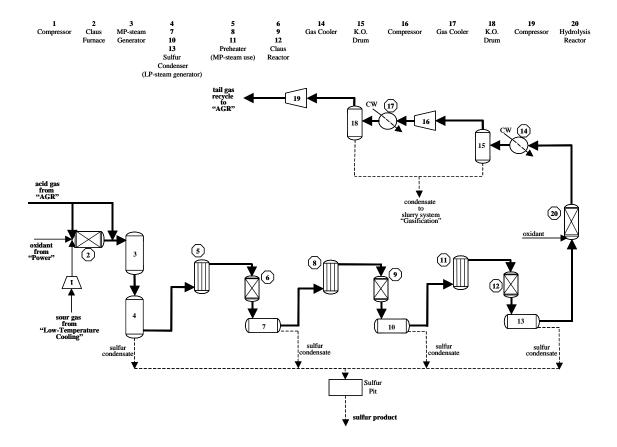


Figure 6.6 – Sulfur Recovery Section - Conventional Methanol Synthesis

	Major constituents (vol%)
H <sub>2</sub>	1.56
CO	0.26
CO <sub>2</sub>	95.53
H <sub>2</sub> O	0.44
N <sub>2</sub>	0.79
Ar	0.06
$H_2S$	1.26
SO <sub>2</sub>	0.00
COS	0.00
NH <sub>3</sub>	0.07
HCN	0.00
Methanol	0.00
Total	99.97

 Table 6.18 - Recycle Tail Gas Composition - Conventional Methanol Synthesis

## **Power Section**

The Power Section process flow diagram is shown in Figure 6.7. This is identical to the process flow diagram for the conventional IGCC Power Section, except that a purge gas stream from the Methanol Synthesis Section and the clean fuel gas stream are both expanded and mixed before being fired in the gas turbine combustors. An oxygen stream, at 108,567 kg/hr (239,345 lb/hr), with 95% purity, is generated by conventional, pressurized, cryogenic air separation (ASU). High-pressure fuel gas from the fuel gas cleaning process, at 130,988 kg/hr (288,774 lb/hr), 271°C (520°F) and 6,846 kPa (993 psia) is expanded (Item 14) to the pressure needed by the turbine combustors, and electric power is generated. Purge gas from the methanol synthesis plant, at 94,222 kg/hr (207,720 lb/hr), 32°C (89°F) and 6,440 kPa (934 psia) is expanded (Item 20) and is combined with the clean fuel gas. Low-purity nitrogen from the ASU is humidified (Item 25) to about 15 vol% water vapor using low-grade heat sources, and the nitrogen stream, at 333,848 kg/hr (735,997 lb/hr) is mixed with the clean fuel gas before the mixture is distributed to the gas turbine combustors (Item 6).

The turbine combustors, advanced, low-NOx burners specifically designed for low heating-value fuel gas, operate with an outlet temperature of about 1524°C (2775°F), and with the peak flame temperature of less than 1649°C (3000°F). The NOx emission is expected to be less than 5 ppmv (dry, corrected to 15% oxygen). The turbine exhaust gas has a mass flow of 1,727,969 kg/hr (3,809,456 lb/hr) and a temperature of 621°C (1150°F). The exhaust gas passes through the heat recovery steam generator (Item 9), generating a superheated, high-pressure steam flow of 401,890 kg/hr (886,000 lb/hr). The fates of the contaminants in the Power Section are listed in Table 6.19. The stack gas from the power plant has a temperature of 104°C (220°F) and a composition listed in Table 6.20.

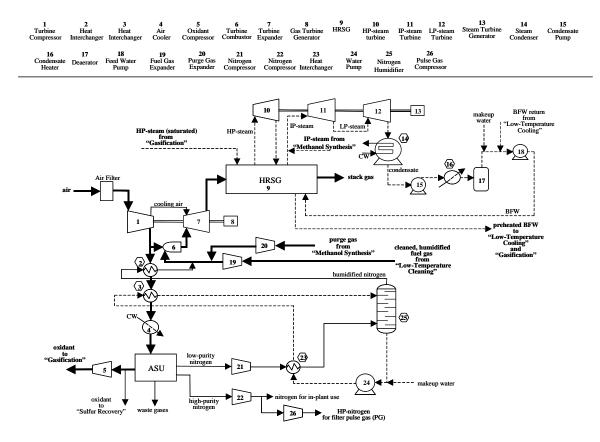


Figure 6.7 – Power Section - Conventional Methanol Synthesis

Contaminant	Removal	Contaminant form conversion
	(% of inlet to Section)	
Sulfur species	0	Oxidation to SOx
Halides	NA	
Ammonia	0	partial oxidation conversion to NOx
Hydrogen cyanide	NA	
Mercury	0	partial conversion to oxidized forms

	Major constituents (vol%)
O <sub>2</sub>	7.92
CO <sub>2</sub>	10.12
H <sub>2</sub> O	10.65
N <sub>2</sub>	70.46
Ar	0.85
Total	100.00
	Minor contaminants
SO <sub>2</sub> (ppmv)	0.033
NOx (ppmv)	5
Mercury (ppbv)	0.012
Particulate (ppmw)	0.1

 Table 6.20 - Stack Gas Composition - Conventional Methanol Synthesis

## 6.2 CONVENTIONAL METHANOL SYNTHESIS PLANT PERFORMANCE ESTIMATES

The breakdown of power generation and power use in the Methanol Synthesis plant with conventional, low-temperature gas cleaning technology is shown in Table 6.21. The Power Island of the plant generates a net 348.5 MWe which includes 8.6 MWe from the fuel gas and purge gas expanders. The ASU consumes a total of 41.2 MWe of power. The next dominant power consumer in the plant is the AGR Section, 12.8 MWe consumed primarily for gas refrigeration. The net plant efficiency of 32.7% (HHV) does not include any credit for the methanol product heating value.

Table 6.22 lists several quantities related to the use of resources and emissions in the plant with conventional, low-temperature gas cleaning technology. The plant uses a large quantity of LP-steam in the gas cleaning process steps. Cooling water use in the processing steps is also very large. Large quantities of process condensate are generated, primarily in the Low-Temperature Cooling Section. A moderate level of fresh process water is required in the gas cleaning processes. A large quantity of boiler-quality makeup water for fuel gas humidification is used.

The total sulfur emissions are expressed under three different bases: percent removal, lb per unit fuel energy input, and mass per MWe net power generated. The sulfur emissions are extremely low, and are comparable to the sulfur emissions from a natural gas-fired, combined-cycle power plant. Estimated solid waste rates from the plant are large, resulting mainly from the wet slag product and the waste salts generated. The rate of solid waste is not sensitive to the gas cleaning process performance.

Section	Power (MW)			
Power Island Generation				
Turbine air compressor	-180.3			
Gas turbine expander	371.4			
Steam turbine cycle	161.7			
Fuel gas expander	6.2			
Purge gas expander	2.4			
Generator	-7.2			
BOP	-5.7			
Total power generation	348.5			
Air Separation Consumption				
ASU Air compressor	0			
Oxygen compressor	8.2			
Nitrogen compressor	9.1			
ASU	23.9			
Total power use	41.2			
Gasification Consumption				
Compressors	0.3			
Pumps	0.2			
Coal handling and preparation	1.8			
Ash handling	1.5			
Total power use	3.8			
Low-Temperature Cooling Consumption				
Pumps	0.0			
AGR Consumption				
Refrigeration	10.9			
Compressors	1.3			
Pumps	0.6			
Total power use	12.8			
Sulfur Recovery Consumption				
Compressors	2.3			
Pumps	0.0			
Total power use	2.3			
Methanol Synthesis Consumption				
Pumps	0.0			
Total Plant				
Net plant power generated, MW	288.3			
Plant net heat rate, kJ/kWh (Btu/kWh) (HHV)	11,008 (10,434)			
Plant net efficiency, %, (LHV)	35.2			
Plant net efficiency, %, (HHV)	32.7			

 Table 6.21 – Methanol Synthesis Plant Thermal Performance with Conventional Gas Cleaning

Process Steam & Water	
Net IP steam process use, MJ/hr (10 <sup>6</sup> Btu/hr)	-264 (-2.5)
Net LP steam process use, MJ/hr (10 <sup>6</sup> Btu/hr)	77,233 (73.2)
Total process cooling water use,	308,089 (292)
MJ/hr (10 <sup>6</sup> Btu/hr)	
Net process condensate generated, kg/hr (lb/hr)	2,641 (5,823)
Total process water used, kg/hr (lb/hr)	3,139 (6,920)
Net process water makeup, kg/hr (lb/hr)	498 (1,097)
Total boiler-feed-water makeup, kg/hr (lb/hr)	59,972 (132,213)
Emissions	
Sulfur total removal efficiency, %	99.9985
Sulfur total emission (HHV),	0.0426 (9.916x10 <sup>-5)</sup>
mg/MJ (lb SO <sub>2</sub> / $10^6$ Btu)	
Sulfur total emission, kg/MW (lb SO <sub>2</sub> /MW)	0.00044 (0.00096)
NOx total emission (HHV),	4.30 (0.0100)
mg/MJ (lb NO <sub>2</sub> / $10^6$ Btu)	
Particulate emission, mg/MJ (lb/ 10 <sup>6</sup> Btu) (HHV)	0.0071 (1.65E-05)
Hg emission, mg/MJ (lb/TBtu) (HHV)	0.043 (0.101)
Chemicals, Sorbents, and Catalysts, kg/hr	
(lb/hr)	
Methanol	136 (300)
Mercury sorbent	2.3 (5)
Guard bed sulfur sorbent	0.9 (2)
Guard bed metal carbonyl sorbent	0.9 (2)
COS hydrolysis catalyst	0.5 (1)
Claus reactor catalyst	2.3 (5)
Solid waste, kg/hr (lb/hr)	
Slag product (25 wt% water)	16,313 (35,964)
Waste salts (25 wt% water)	338 (745)
Sorbent & catalyst wastes (hazardous)	7 (15)
Total solid waste	16,658 (36,724)

 Table 6.22 – Methanol Plant Conventional Gas Cleaning Resource Use and Emissions

# 6.3 CONVENTIONAL METHANOL SYNTHESIS PLANT COST ESTIMATES

No financial analysis for this co-production plant has been performed in this evaluation. The evaluation utilizes direct comparison of gas cleaning equipment investment, annual operating cost of the gas cleaning equipment, total plant electricity production and methanol production as the basis for comparison.

The major equipment purchase costs and installed costs are listed in Table 6.23 for each of the cleaning sections of the plant evaluated. The Gasification Section's cost for the convective cooler has been included in the cost breakdown since this will differ from the convective cooler cost in the Novel Gas Cleaning process. Also, the Mercury Removal cost has been taken out of the Low-Temperature Cooling Section cost and reported as a separate item. Only the gas cleaning costs associated with the Methanol Synthesis Section are included. The Low-temperature Cooling Section is the second most expensive of the gas cleaning sections, and its cost approaches the cost of the Desulfurization Section. The total gas cleaning costs are also reported on the basis of dollars per kilowatt of net power generated. While the total gas cleaning equipment cost approaches 200 \$/kW, this is clearly only a small portion of

the total plant equipment cost. The impact of gas cleaning on the overall plant performance and its operating cost are much more important factors.

Plant Section	Cost, k\$			
Raw gas convective cooling				
purchased equipment	1,481			
installed equipment	2,962			
Low-temperature Cooling				
purchased equipment	6,824			
installed equipment	13,294			
Mercury removal				
purchased equipment	481			
installed equipment	704			
AGR				
purchased equipment	11,177			
installed equipment	19,385			
Sulfur Recovery				
purchased equipment	6,345			
installed equipment	10,781			
Methanol Synthesis (gas cleaning only)				
purchased equipment	1,766			
installed equipment	3,443			
Fuel gas and Purge gas expanders				
purchased equipment	870			
installed equipment	1,305			
Total				
purchased equipment	28,944			
installed equipment	51,873			
purchased equipment, \$/kW	96			
installed equipment, \$/kW	172			
Total Plant				
TCR, k\$	516,316			
TCR,\$/kW	1791			

Table 6.23 - Conventional Gas Cleaning Technology Equipment Cost Breakdown

The Total Capital Requirement for the Methanol Synthesis plant using conventional, lowtemperature gas cleaning technology was estimated by scaling the non-gas cleaning equipment costs from IGCC power plant cost data. A confirmed basis does not exist for making this estimate, so the total plant costs are uncertain, but represent a good basis for technology comparisons. Table 6.24 shows the estimate for the Total Capital Requirement for the plant, and lists the breakdown for the Cost-of-Electricity (COE) for the plant using representative by-product values for the methanol and elemental sulfur products. Coal slag is assumed to be a disposal product having no value. The objective here is not to assess the financial implications for such a plant, but it to provide a basis for comparison with the COE for a comparable plant using Novel Gas Cleaning technology.

	COE
	(Cents/kWh)
Fixed O&M	0.40
Variable O&M	0.26
Consumables	0.22
water	0.040
chemicals, sorbents, catalysts	0.051
waste Disposal	0.130
By-product credit	1.10
sulfur	0.073
methanol	1.03
Fuel	1.94
Capital charges	3.83
Total	5.55

Table 6.24 – Methanol Plant with Conventional Gas Cleaning -Cost-of-Electricity

Table 6.24 shows clearly that the gas cleaning process equipment cost has little impact on the COE of the plant, but the gas cleaning process influence on the plant efficiency is very important. Consumables in the conventional technology plant have a very small impact on the COE. This sensitivity perspective indicates that the gas cleaning process focus should be on minimizing performance losses rather than on minimizing equipment costs.

## 7. METHANOL SYNTHESIS WITH NOVEL GAS CLEANING

The Novel Gas Cleaning technology for methanol synthesis is similar to the Novel Gas Cleaning technology applied for IGCC with Future Standards (Section 5), but an additional syngas polishing section is used to meet the methanol synthesis requirements. This section of the report describes the Novel Gas Cleaning technology applied to methanol synthesis. The performance and cost-potential of methanol synthesis using Novel Gas Cleaning technology is compared to that of methanol synthesis using conventional gas cleaning technology, as described in Section 6.

# 7.1 DESCRIPTION OF METHANOL SYNTHESIS WITH NOVEL GAS CLEANING

The overall Methanol Synthesis process is illustrated in Figure 7.1. It is similar to the IGCC power plant using Novel Gas Cleaning technology with Future Standards, except that

- a greater coal feed rate, about 16% greater, is used to accommodate the fuel needs for both power generation and methanol synthesis,
- the gasifier is operated at a much higher pressure to generate syngas that can meet the high-pressure needs of the methanol synthesis reactor,
- the partially cleaned gas is split into a fuel gas stream for power generation and a syngas stream for methanol synthesis, and this syngas stream is polished using a water scrubbing process to meet its stringent cleaning standards.

The plant consists of six sections: Gasification, Fuel Gas Cleaning, Sulfur Recovery, Syngas Cleaning, Methanol Synthesis & Distillation, and Power. As with the conventional methanol synthesis plant described in Section 6, a stream of "syngas" is split from the cleaned fuel gas stream, and this is further cleaned of sulfur, halides and fuel-bound nitrogen using a wet scrubbing process before the syngas is conditioned and reacted for methanol synthesis. The methanol synthesis purge gas is expanded and combined with the fuel gas fed to the Power Section of the plant.

The very stringent gas cleaning standards for methanol synthesis are met by using a zinc oxide sorbent in a regenerative, transport reactor system for bulk sulfur control, followed by two stages of oncethrough, zinc-based sorbent polishing. Halides are controlled by once-through nahcolite sorbent injection into a primary filter vessel located before the bulk sulfur removal process, followed by an additional polishing stage of halide wet scrubbing at a lower temperature condition. For methanol synthesis, specifications for ammonia and HCN are also very stringent. Because no ammonia sorbent is current available, and because there is little advantage in retaining water in the synthesis gas stream, HCN hydrolysis to ammonia is followed by warm-water scrubbing of ammonia. Using this scrubbing scheme, the ammonia is easily reduced to less than the specified level for methanol synthesis, and the halides are simultaneously reduced to acceptable levels. Condensate containing ammonia and HCl is recycled to the gasifier where ammonia is decomposed. Recycled HCl is eventually captured in the Bulk Halide Removal system. Metal carbonyls are less likely to form in the Novel Gas Cleaning process than in the conventional gas cleaning process, but because of uncertainty, a carbonyl guard bed is included in the Novel Gas Cleaning process.

## **Gasification Section**

Figure 7.2 shows the process flow diagram constructed for the Gasification Section using the Novel Gas Cleaning technology, and includes designation of the Coal Receiving and Handling System and the Slurry Preparation System. The process diagram is nearly identical to the Gasification Section process flow diagram for IGCC with Future Standards in Section 5. Oxidant from the Power Section's Air Separation Unit, at about 106,476 kg/hr (234,735 lb/hr) combines with the coal slurry, at 166,004 kg/hr (365,969 lb/hr) to generate high-temperature, raw gas and slag streams. The raw gas flow rate at the exit of the Convective Cooler (Item 7) is about 273,006 kg/hr (601,864 lb/hr), with heat content of 2,254,745 MJ (2,137 x 10<sup>6</sup> Btu/hr) (LHV) and heating value of about 7.78 MJ/Nm<sup>3</sup> (198 Btu/scf), and

includes about 11,794 kg/hr (26,000 lb/hr) of recycled, clean fuel gas used as soot blower gas for the raw gas heat exchangers.

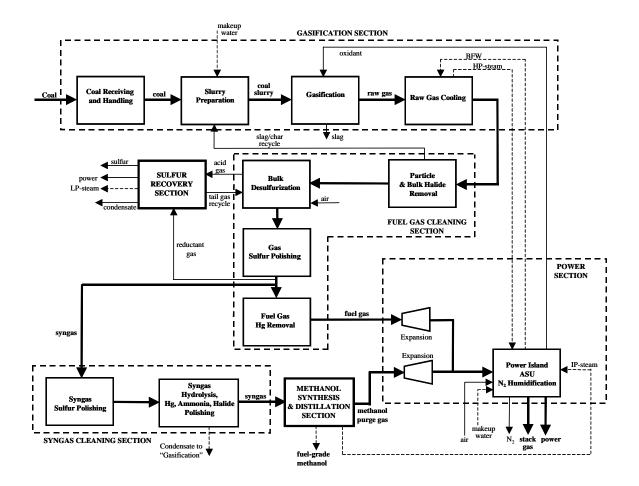


Figure 7.1 - Overall Plant Scheme for Methanol Synthesis with Novel Gas Cleaning

The raw gas exits the gasifier at about 1407°C (2565°F) and 8,791 kPa (1275 psia), and 593°C (1100°F) from the Convective Cooler, with the estimated composition listed in Table 7.1, not including entrained slag. The raw gas is cooled in a radiant cooler, raising saturated, high-pressure (HP) steam, and cooling the fuel gas to about 816°C (1500°F) to solidify slag particles before cooling the fuel gas further in a convective cooler to generate additional saturated HP-steam.

# **Fuel Gas Cleaning Section**

Figure 7.3 shows the process flow diagram for the Fuel Gas Cleaning Section. It is identical to Figure 5.3, for the Current Standards case, except that a Fuel Gas Polishing Filter-Reactor (Item 16) and Polishing Sulfur Sorbent Feed System (Item 15), and Sorbent Waste Removal System (Item 17) have been inserted after the bulk desulfurizer for additional sulfur removal. The flows and compositions are also similar except as noted below.

	Major constituents (vol%)
H <sub>2</sub>	25.39
CH <sub>4</sub>	0.30
СО	40.77
CO <sub>2</sub>	9.86
H <sub>2</sub> O	20.68
N <sub>2</sub>	1.80
Ar	0.14
Total	98.94
	Major contaminants (ppmv)
$H_2S$	8,515
COS	636
$CS_2$	0.6
S <sub>X</sub>	2
SO <sub>2</sub>	1
NH <sub>3</sub>	687
HCN	20
HCl	436
Hg (ppbv)	3

Table 7.1 - Raw Gas Composition – Novel Gas Cleaning



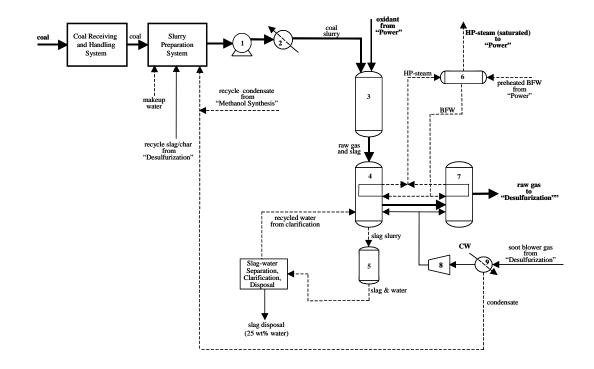


Figure 7.2 - Gasification Section with Novel Gas Cleaning

<u>Particle & Bulk Halide Removal</u>: This section is identical its functions and performance as the Particle & Bulk Halide Removal system described in Section 5 for IGCC. It operates at 593°C (1100°F) and removes raw gas particulate using a conventional cyclone followed by a ceramic barrier filter-reactor, as was used for IGCC in Section 5. 2,424 kg/hr (5,343 lb/hr) of a halide sorbent, nahcolite, is injected into the gas entering the filter-reactor to capture halides. This feed rate is equivalent to a sodium-to-halide molar feed ratio of greater than 4. 99.1% of the halides (HCl-basis) are removed, to a content of about 4 ppmv. Because the gas volumetric flow at this high pressure is relatively small, the filter-reactor is relatively small, less than 9 feet in diameters and 50 feet tall, containing two standard filter clusters, holding a total of 374 standard, low-cost, commercial ceramic filter candles.

<u>Gas heat interchanger (Item 5)</u>: The gas is cooled in a heat interchanger to 482°C (900°F) preheating the methanol purge gas stream that passes to the Power Section of the plant. This gas is then mixed with recycled tail gas from the downstream, sulfur recovery process, a flow of about 11% of the gas stream.

Bulk desulfurization: The gas now enters the bulk-desulfurization process where its total sulfur content is reduced to less than 50 ppmv, about a 99.5% reduction of H<sub>2</sub>S, COS, CS<sub>2</sub>, Sx, and SO<sub>2</sub> contained in the gas, with significant hydrolysis of CS<sub>2</sub>, Sx and SO<sub>2</sub> to H<sub>2</sub>S also expected. The zinc titanate sorbent is assumed to have a Zn/Ti mole ratio of 1.0, and to operate with a net, sorbent makeup stoichiometric molar feed ratio of 0.0027 Zn/S provided by the sorbent feed system D-P-3. The gas passes through a transport reactor (Item 6) of circulating zinc titanate sorbent, producing a bulk-desulfurized gas having total sulfur content of about 43 ppmy and containing some entrained sorbent particles that escape the transport reactor disengaging section. The partially-sulfided sorbent particles circulating in to the desulfurizer leg have a molar ratio for ZnO/S of about 221. The sulfided sorbent particles circulate to the parallel, entrained regenerator vessel (Item 8) where air contacting generates an SO<sub>2</sub> acid gas and regenerated zinc titanate sorbent. Compressed air is provided by compressor Item 12, and a fired heater (Item 11) is also provided for preheating the regenerator air. Nitrogen fluffing gas and nitrogen purging of the transport legs between the gasifier and regenerator are used, this compressed nitrogen coming from the Power Island. The regenerator acid gas, at about 733°C (1352°F), passes through a relatively small barrier filter (Item 9) to separate its entrained sorbent particles. The entrained sorbent particles captured in this filter are cooled and back to the standleg of sorbent flowing back to the desulfurizer vessel, or may be drained into the bulk desulfurized fuel gas exiting the vessel. The bulk-desulfurized gas leaves the process at about 550°C (1022°F) carrying all of the sorbent lost by attrition and elutriation from the bulkdesulfurization process. The bulk desulfurized fuel gas has a flow of 311,534 kg/hr (686,803 lb/hr), at 550°C (1022°F) and 7770 kPa (1127 psia), and its composition is listing in Table 7.2. Note that it contains significant particulate in the form of zinc-titanate sorbent particles. The relatively high nitrogen content in the fuel gas results largely from the sulfur recovery tail gas. The acid gas generated has the composition listed in Table 7.3. Its flow is 28,970 kg/hr (63,867 lb/hr) at 733°C (1352°F) and 7688 kPa (1115 psia). Note that in contrast to the IGCC application in Section 5, for methanol synthesis it is desirable to minimize the nitrogen content of the methanol synthesis gas, and recycled fuel gas us used for pulse gas cleaning, sorbent feeding, fluffing and stripping rather than using nitrogen. Waste sorbent handling continues to use nitrogen since this does not dilute the synthesis gas stream.

<u>Fuel Gas Sulfur Polishing Filter-Reactor</u>: The bulk desulfurized gas is cooled in process heat exchangers 13 and 14 to 482C (900°F). 20 kg/hr (43 lb/hr) of polishing sulfur sorbent particles, also zinc titanatetype, are injected into the gas and, combined with the entrained sorbent particles from the bulkdesulfurization process, the mixture enters a barrier filter and results in additional 96% sulfur removal down to a level of total sulfur less than 2 ppmv. The polished fuel gas composition is listed in Table 7.4. The sorbent makeup rate uses a stoichiometric ratio of 0.2 Zn/S in the regenerative operation. The partially-sulfided sorbent particles carried with the fuel gas have a molar ratio for ZnO/S of about 3.3. The filter separates the entrained bulk-sorbent particles and the polishing sorbent particles from the fuel gas. The collected sorbent particulate is drained from the filter and is pneumatically fed back to the bulk desulfurization process regenerator. This filter-reactor is slightly smaller in dimension than the bulk halide filter-reactor and contains the same number of filter candles. The small amount of waste sorbent ultimately drained from this system is either disposed directly or is fed to the gasifier to be incorporated into the plant slag by-product. At this point the gas stream is split into the "fuel gas" stream, at 162,454 kg/hr (358,144 lb/hr) and the "syngas" stream at 148,688 kg/hr (327,796 lb/hr).

<u>Gas cooling</u>: The fuel gas stream is cooled in Item 18 to a temperature of about 204°C (400°F), suitable for mercury removal in the Fuel Gas Mercury filter-reactor (Item 20). The Hg-cleaned fuel gas passes through a heat interchanger (Item 22) to reheat the clean fuel gas from 204°C (400°F) to 433°C (811°F) and to cool the syngas stream passing to the Methanol Synthesis Section. The clean fuel gas then passes to heat interchanger 13 where it is reheated to 522°C (972°F), before passing to the Power Section.

	Major constituents (vol%)
H <sub>2</sub>	23.80
CH <sub>4</sub>	0.29
СО	38.20
CO <sub>2</sub>	10.82
H <sub>2</sub> O	19.43
N <sub>2</sub>	7.21
Ar	0.19
Total	99.94
	Major contaminants (ppmv)
$H_2S$	39.78
COS	2.84
HC1	3.65
NH <sub>3</sub>	160
HCN	19
Hg (ppbv)	2.5
Particulate (ppmw)	1411

 Table 7.2 - Bulk Desulfurized Gas Composition – Novel Methanol Synthesis

 Table 7.3 - Acid Gas Composition – Novel Methanol Synthesis

	Major constituents (vol%)					
$N_2$	83.80					
Ar	0.95					
$CO_2$	0.03					
H <sub>2</sub> O	1.03					
SO <sub>2</sub>	14.18					
Total	99.99					

Soot-blower gas, at 11,616 kg/hr (25,608 lb/hr), transport and pulse gas, at 15,513 kg/hr (34,200 lb/hr) and Sulfur Recovery reductant gas, at 8,763 kg/hr (19,319 lb/hr) are extracted from the fuel gas stream at this point. This gas is particulate-free, and is compressed at this point. About 5.4% of this fuel gas stream is separated to be used as a reductant in the sulfur recovery process. These are all recycles streams and result in little fuel gas loss.

	Major constituents (vol%)
$H_2$	23.81
CH <sub>4</sub>	0.29
СО	38.21
CO <sub>2</sub>	10.82
H <sub>2</sub> O	19.44
N <sub>2</sub>	7.21
Ar	0.20
Total	99.98
	Major contaminants (ppmv)
$H_2S$	1.8
COS	0.1
HCl	3.6
NH <sub>3</sub>	160
HCN	19
Hg (ppbv)	2.5
Particulate (ppmw)	0.1

 Table 7.4 - Polished Fuel Gas Composition – Novel Methanol Synthesis

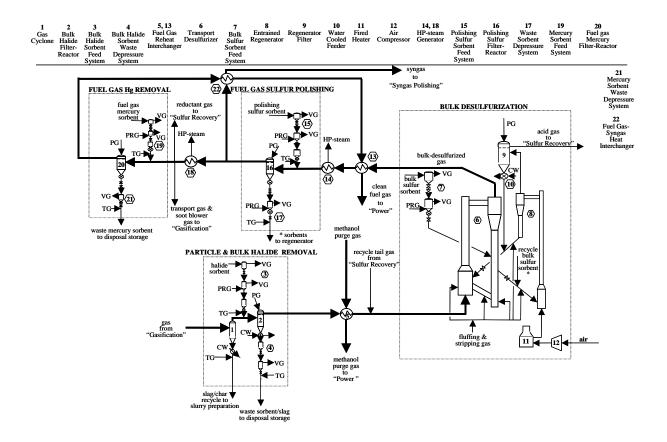


Figure 7.3 - Fuel Gas Cleaning Section - Novel Methanol Synthesis

<u>Fuel Gas Mercury Removal System:</u> The mercury removal process is similar to the previous descriptions for IGCC application, and is designed and operated for 95% mercury removal. The mercury removal process scheme is a once-through operation using powdered mercury sorbent injection into a filter-reactor (Item 20). The mercury sorbent is injected into the fuel gas by feed system (Item 19). The assumed feed rate is 7 kg/hr (15 lb/hr), equivalent to a sorbent-to-mercury mass ratio greater than 2000. The cooled fuel gas passes into a ceramic candle barrier filter (Item 20) to conduct mercury removal. This small filter-reactor vessel is slightly greater than 1.5 m (5 ft) in diameter, with a total height of 13.1 m (43 ft), and holds 187 filter elements. The filter also collects any equipment-corrosion particulate that might be present in the fuel gas from upstream sources. The mercury sorbent is drained from the filter and is depressurized (Item 21) to be disposal as a hazardous solid.

An alternative Fuel Gas Cleaning process with a simpler configuration having lower equipment cost is shown in Figure 7.4. Here the sulfur polishing filter-reactor and the fuel gas mercury removal filter-reactor are combined (Item 17) and are placed to follow the fuel gas coolers. This single filter-reactor, operating at a temperature of 204 to 288°C (400 to 550°F) would accomplish both gas cleaning functions with a mixture of two injected sorbents. The once-through sorbents would be depressurized and stored for disposal, and there would be no recycle of sulfur sorbent to the bulk sulfur removal system. While this alternative configuration must be considered for future development, defining an additional set of operating conditions for the candidate mercury and polishing sulfur sorbents, its specific performance and cost have not been estimated in this evaluation.

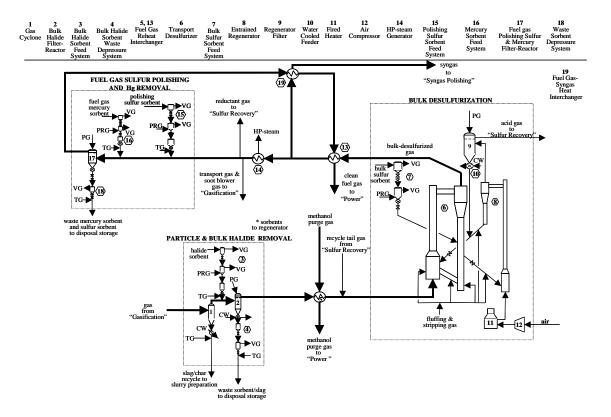


Figure 7.4 – Alternative Fuel Gas Cleaning Section - Novel Methanol Synthesis

Contaminant	Removal	Contaminant conversions
	(% of inlet to Section)	
Sulfur species	99.98	Partial hydrolysis
		Combustion to SOx
		Sorbent fixation
Halides	99	Sorbent fixation
Ammonia	75	Catalytic partial decomposition to nitrogen
Hydrogen cyanide	0	
Mercury	95	Sorbent fixation

 Table 7.5
 – Fate of Contaminants in Fuel Gas Cleaning Section - Novel Methanol Synthesis

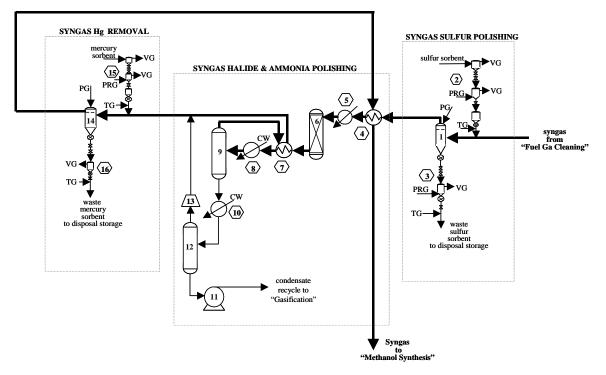
<u>Comments on the Mercury Removal process</u>: The performance of the filter-reactors with injected sorbents have not yet been experimentally established, so the process evaluation is speculative. In particular, the type of mercury sorbent, the characteristics of the mercury removal process, and the performance of the mercury removal process are all highly speculative at this time. The evaluation identifies the process' acceptable range of operating conditions, required range of performance, and potentially acceptable operational modes:

- mercury removal should operate as hot as about 204-288°C (400-550°F), removing 90-98% of the mercury,
- the type of mercury sorbent has not been established, and it could be either a once-through or a regenerative sorbent -- it is expected that it will be advantageous for it to be a regenerative adsorbent (e.g., a zeolite),
- the mercury removal process should be a continuous process -- it is expected that a continuous process will have advantages over a batch process with respect to power plant availability and performance,
- the selected mercury sorbent must not result in the release of any contaminants, such as sulfur, to the cleaned fuel gas that will exceed the emission requirements,
- the mercury adsorbent, if regenerative, might be regenerated by heating it in an available, clean gas or vapor stream, such as nitrogen or steam, to a temperature of no greater than 343°C (650°F), with liquid mercury being subsequently condensed and separated,
- the mercury removal step provides the final, clean fuel gas that goes to the gas turbine combustors, and it should have the capability of also handling upstream equipment corrosion particulate removal,
- the minimum operating temperature for the mercury removal stage is about 204°C (400°F), based on vapor condensation -- if lower operating temperatures are desired, water vapor will be condensed from the fuel gas,
- if water vapor in the fuel gas hinders the mercury removal step, the fuel gas can be reduced significantly in water vapor content by adding a water gas shift reaction stage,

# **Syngas Polishing Section**

The scheme for syngas halide and ammonia polishing control considered is based on warm-water scrubbing of the syngas. The process flow diagram for the Syngas Polishing Section using water scrubbing is shown in Figure 7.5. This process removes halides and ammonia to very low levels and generates a water stream used in the coal slurry stream that is fed to the gasifier. The contained ammonia in the scrub water is decomposed in the gasifier and the contained halides are ultimately recycled and captured by the bulk halide removal process. The warm-water scrubbing process can reduce ammonia levels in the fuel gas to less than 10 ppmv, and halides to negligible levels.

1 Syngas Sulfur Filter-Reactor	2 Sulfur Sorbent Feed System	3 Sulfur Sorbent Waste Depressure System	<b>4, 7</b> Syngas Heat Interchanger	5 Syngas Cooler	6 Hydrolysis Reactor	8 Syngas Condenser	9 Condensate Separator	10 Condensate Cooler	11 Condensate Pump	12 Flash Vessel	13 Compressor	14 Syngas Mercury Filter-Reactor	15 Mercury Sorbent Feed System	16 Mercury Sorbent Waste Depressure System
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**Figure 7.5 – Syngas Polishing Section - Novel Methanol Synthesis** 

<u>Sulfur Polishing</u>: A barrier-filter reactor (Item 1) operated at 288°C (550°F) is used to reduce the syngas sulfur content to the required level, injecting 6 kg/hr (13 lb/hr) of a Zn-based sulfur sorbent into the gas using injection system (Item 2). The Zn/S molar feed ratio in this system is about 4.7. The same Zn-based sorbent used in the Bulk Sulfur Removal system, crushed to a finer size distribution, is used here. The filter-reactor used here has about 1.8 m (5.5 ft) diameter and is 13.1 m (43 ft) tall, similar in design to the fuel gas polishing filter-reactor.

<u>Wet Scrubbing</u>: The sulfur-polished syngas HCN content is hydrolyzed at 204°C (400°F) to ammonia in a catalytic reactor (Item 6) after gas cooling by a heat interchanger (Item 4) and cooler (Item 5). The hydrolyzed gas is then recuperatively cooled (Item 7), and then cooled and condensed further in Item 8 to 101°C (213°F). The condensate-gas mixture passes through a gas-condensate separator (Item 9), simultaneously absorbing halides and ammonia into the condensate to very low levels. The separated syngas is reheated to 188°C (370°F) across Item 7. The collected condensate is cooled further to 38°C (100°F) across cooler 10, and is then flashed to 117 kPa (17 psia), separating out a condensate stream that is recirculated to the gasification slurry preparation system. The offgas is compressed (Item 13) and placed back into the syngas.

<u>Mercury Removal</u>: Mercury sorbent is injected at 4.5 kg/hr (10 lb/hr) into the syngas (Item 15), and the gas-sorbent mixture passes through the mercury filter-reactor (Item 14), removing 95% of the syngas mercury. This filter-reactor is about 5 feet in diameter and 43 feet tall, operating with a face velocity of about 1.2 m/min (3.9 ft/min). The mercury sorbent is depressurize (Item 16) for storage and disposal. A carbonyl guard bed (Item 17) could also be inserted into the process, but should not be needed with

proper selection of materials of construction. The cleaned syngas is reheated across a heat interchanger (Item 4). The cleaned syngas composition is estimated in Table 7.6.

	Major constituents (vol%)
$H_2$	29.13
$CH_4$	0.36
CO	46.75
$CO_2$	13.22
H <sub>2</sub> O	1.45
$N_2$	8.86
Ar	0.24
Total	100.00
	Major contaminants (ppmv)
$H_2S$	0.47
COS	0.00
HC1	0.00
NH <sub>3</sub>	5.5
HCN	0.03
Hg (ppbv)	0.061

Table 7.6 – Syngas Composition – Novel Methanol Synthesis

An alternative Syngas Polishing Section process configuration with low-cost potential is illustrated in Figure 7.6. The syngas sulfur polishing and mercury removal functions are combined into a single filter-reactor in this configuration, simplifying the configuration greatly. The combined filter reactor could be placed before the wet scrubber, operating at about 204°C (400°F), and could be placed to follow the wet scrubber, operating at a temperature as low as 104°C (220°F). This then defines the range of operating temperatures needed for the combined polishing sulfur sorbent and the syngas mercury sorbent if this alternative configuration is to be used: 104-204°C (220 – 400°F). An additional simplification is to eliminate HCN hydrolysis, with the expectation from the literature than HCN is not really a significant contaminant to the methanol catalyst in the Liquid Phase Methanol process.

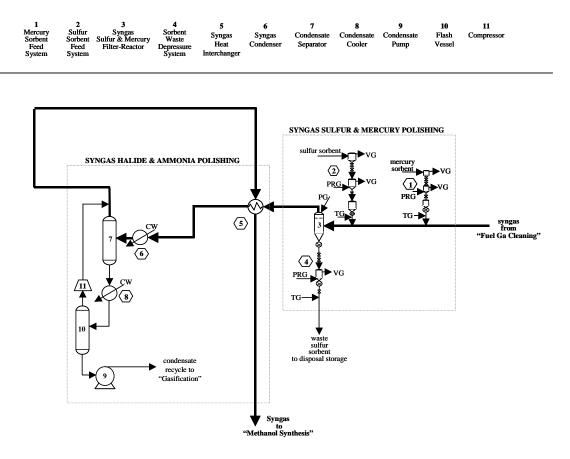


Figure 7.6 – Alternative Syngas Polishing Section - Novel Methanol Synthesis

The fate of the contaminants within the Syngas Polishing section are listed in Table 7.7. The performance hypothesized to be achieved here satisfies all of the methanol synthesis gas cleaning requirements without additional guard beds inserted before the synthesis reactor.

Contaminant	Removal	Contaminant conversions
	(% of inlet to Section)	
Sulfur species	98	Sorbent fixation
Halides	100	Water scrubbing
		Recycle to Bulk Halide
		Removal
Ammonia	97	Hydrolysis
		Water scrub
		Decomposition in gasifier
Hydrogen cyanide	100	Hydrolysis
Mercury	98	Sorbent fixation

Table 7.7 – Fate of Contaminants in Syngas Polishing Section - Novel Methanol Synthesis

## **Methanol Synthesis Section**

The Methanol Synthesis Section flow diagram is shown in Figure 7.7. The first processing step is to humidify the syngas to about 8.5 vol% in Item. The humid syngas passes through the methanol synthesis reactor where about 41% of the syngas hydrogen is converted to methanol. LP-steam and IP-steam are generated in the methanol reactor fluid bed, controlling the exit gas temperature to about 249°C (480°F). A cyclone captures and recycles elutriated catalyst particles and oil to the bed. The methanol reactor product gas is then cooled to about 99°C (210°F) and oil is separated from the product gas in a separation vessel. The product gas is further cooled to about 38°C (100°F) before passing through a knock-out vessel to separate the synthesis liquid and purge gas products. The purge gas, at 124,280 kg/hr (273,985 lb/hr) and 6,433 kPa (933 psia), from the knock-out vessel is reheated to about 482°C (900°F) before passing to the Power Section.

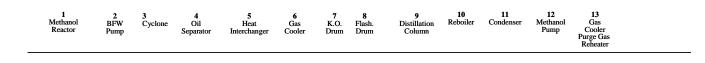
The purge gas composition is presented in Table 7.8, and has a heating value of about 8.73 MJ/Nm<sup>3</sup> (222 Btu/scf). The synthesis product, at 11,816 kg/hr (26,050 lb/hr), is flashed to about 117 kPa (17 psia) to separate some dissolved gases, about 107 kg/hr (235 lb/hr) from the crude methanol product. The crude methanol is distilled to generate the fuel-grade methanol product, at 8998 kg/hr (19,837 lb/hr). In total, 91.3 % of the methanol generated in the synthesis reactor is recovered in the final product. The product methanol composition is presented in Table 7.9

	Major constituents (vol%)
$H_2$	29.58
$CH_4$	0.39
CO	35.55
$CO_2$	24.05
H <sub>2</sub> O	0.01
N <sub>2</sub>	9.69
Ar	0.26
Methanol	0.45
Total	99.98

Table 7.8 – Purge Gas Composition - Novel Methanol Synthesis

 Table 7.9 – Fuel-Grade Methanol Composition - Novel Methanol Synthesis

	Major constituents (wt%)
H <sub>2</sub> O	0.41
$CO_2$	0.05
Methanol	98.20
Methyl formate	0.15
Ethanol	1.18
Total	99.99



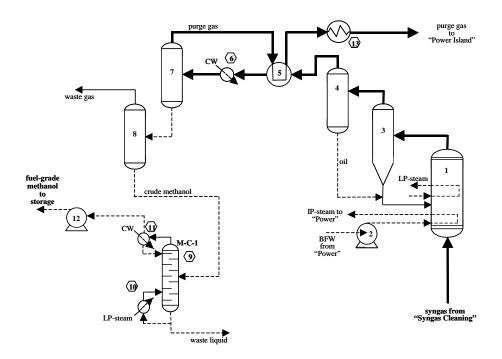


Figure 7.7 – Methanol Synthesis Section - Novel Methanol Synthesis

## **Sulfur Recovery Section**

Figure 7.8, the process flow diagram for the Sulfur Recovery Section, is identical to Sulfur Recovery in the IGCC power plant. The acid gas from the bulk desulfurizer regenerator contains about 14 vol% SO<sub>2</sub>, with very low oxygen content, and the remaining components being largely nitrogen with a little CO<sub>2</sub>. This acid gas is first expanded (Item 1) to about 193 kPa (28 psia), cooling the gas to about 224°C (435°F) and generating a small amount of electrical power. The acid gas is then mixed with a portion of bulk desulfurized fuel gas and is catalytically reacted to hydrogenate an appropriate portion of the acid gas SO<sub>2</sub> to H<sub>2</sub>S for the Claus reaction in reactor Item 2. The gas is then cooled in a boiler (Item 3) to generate IP-steam, followed by a boiler-sulfur condenser (Item 4) generating LP-steam. Any elemental sulfur contained in the product acid gas is separated before the gas enters the first Claus reactor (Item 5). The remaining steps of the process are very similar to those described for the conventional fuel gas cleaning sulfur recovery process: three stages of gas preheat, Claus reactors and sulfur condensers.

The sulfur recovery process recovers about 95.7% of the sulfur content of the acid gas. The collected sulfur streams are combined and the sulfur, at 4,030 kg/hr (8,885 lb/hr) is stored for treatment and marketing as a by-product. The tail gas, at 30,759 kg/hr (67,811 lb/hr), contains a considerable content of sulfur species ( $H_2S$  and  $SO_2$ ), and the only way the power plant can achieve its overall 99.85% sulfur removal goal is for this tail gas to be recompressed and recycled to the bulk desulfurizer. After cooling and compression and condensate removal, the tail gas recycled to the bulk desulfurizer has the composition reported in Table 7.10.

	Major constituents (vol%)
H <sub>2</sub>	0.13
CH <sub>4</sub>	0.12
СО	0.21
$CO_2$	20.08
H <sub>2</sub> O	0.35
N <sub>2</sub>	77.62
Ar	0.93
Total	99.47
	Major contaminants (ppmv)
$H_2S$	3772
SO <sub>2</sub>	1677
COS	0
$CS_2$	0
NH <sub>3</sub>	46
HCN	8

 Table 7.10 - Recycle Tail Gas Composition - - Novel Methanol Synthesis

# The fate of the contaminants within the Sulfur Recovery section are listed in Table 7.11.

Contaminant	Removal	Contaminant conversions
	(% of inlet to Section)	
Sulfur species	95.66	Claus conversion to elemental
		sulfur
		Hydrolysis to reduced forms
Halides	NA	
Ammonia	30.0	Furnace decomposition
Hydrogen cyanide	NA	
Mercury	NA	

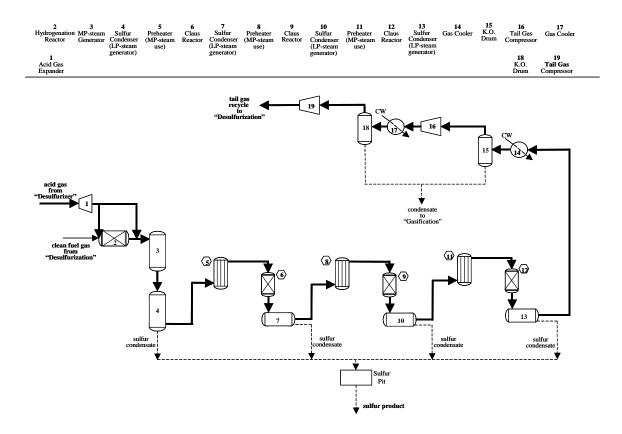


Figure 7.8 – Sulfur Recovery Section - Novel Methanol Synthesis

# **Power Section**

The Power Island process flow diagram is identical to those described for the IGCC cases, except that the clean fuel gas is expanded to the required turbine combustor pressure, and is mixed with expanded methanol purge gas. The process diagram is shown in Figure 7.9. An oxygen stream at 106,476 kg/hr (234,735 lb/hr), with 95% purity, is generated by conventional, pressurized, cryogenic air separation unit (ASU). A relatively low-purity N<sub>2</sub> stream is also produced that is used for clean fuel gas dilution. A smaller stream of high-purity  $N_2$  (99.9%) is also produced that is used in the gas cleaning process for solids pressurization, stripping, purging, pneumatic transport, and filter pulse cleaning. The purge gas stream from Methanol Synthesis, and the clean fuel gas stream are both expanded, recovering electrical power, and are mixed together as a single turbine fuel gas stream. Low-purity nitrogen is humidified to about 16 vol% water vapor using low-grade heat sources, and the nitrogen stream, at 305,726 kg/hr (674,000 lb/hr) is mixed with the clean fuel gas before the mixture is distributed to the gas turbine combustors. The turbine combustors, advanced, catalytic, and/or diffusion flame burners that promote the decomposition of the remaining ammonia in the fuel gas with less than 5% conversion to NOx, operate with an outlet temperature of about 1521°C (2770°F), and with the peak flame temperature of less than 1649°C (3000°F), the NOx emission is expected to be less than 5 ppmv (dry, corrected to 15% oxygen).

The turbine expansion gas has a mass flow of 1,742,665 kg/hr (3,841,854 lb/hr) and a temperature of 619°C (1147°F). The expansion gas passes through the heat recovery steam generator (HRSG), generating a superheated, high-pressure steam flow of 400,529 kg/hr (883,000 lb/h). The stack gas from the power plant has a temperature of 104°C (220°F) and a composition listed in Table 7.12. The fate of the contaminants within the Power section are listed in Table 7.13.

	Major constituents (vol%)	
O <sub>2</sub>	8.27	
$CO_2$	9.99	
H <sub>2</sub> O	10.73	
N <sub>2</sub>	70.16	
Ar	0.85	
Total	100.00	
	Major contaminants	
$SO_2(ppmv)$	0.2	
NOx (ppmv)	5	
Mercury (ppbv)	0.0125	
Particulate (ppmw)	0.1	

 Table 7.12
 - Stack Gas Composition – Novel Methanol Synthesis

IP-steam Turbing Air Cooler Gas Turbine Generator HP-steam turbine LP-steam Turbine Condensate Pump Turbine Compress Heat Heat terchange Turbine Expander Steam Oxidant Compresso HRSG Turbine Combusto Steam Turbine Generator Fuel Gas Expander Purge Gas Expander Nitrogen Compressor Nitrogen Humidifier 17 18 Deaerator Feed Water Pump 22 23 Nitrogen Heat Compressor Interchanger Water Pump Condensate Heater

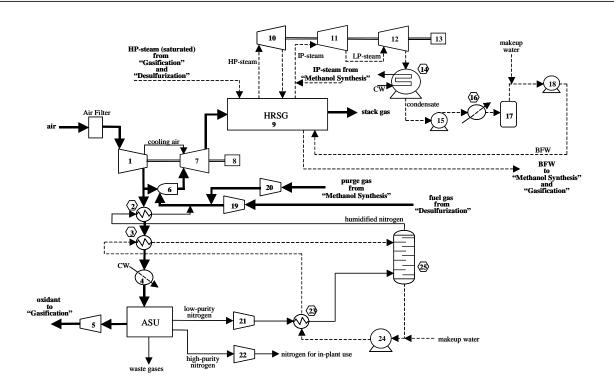


Figure 7.9 – Power Section - Novel Methanol Synthesis

Contaminant	Removal	Contaminant form conversion
	(% of inlet to Section)	
sulfur species	0	Oxidation to SOx
Ammonia	0	partial oxidation conversion to NOx
Mercury	0	Partial conversion to oxidized forms

# 7.2 PERFORMANCE OF METHANOL SYNTHESIS WITH NOVEL GAS CLEANING

The breakdown of power generation and power use in the Methanol Synthesis plant with Novel Gas Cleaning technology is shown in Table 7.14. Note that the Sulfur Recovery Section net power use is nearly zero, with the acid gas expander's generation balancing the power losses in the system.

Section	Power (MW)
Power Island	
Turbine Air Compressor	-182.14
Gas turbine	375.82
Steam turbine	161.17
Fuel gas expander	8.95
Syngas expander	6.60
Generator	-7.4
BOP	-5.6
Gross power	357.4
Air Separation	
ASU Air compressor	0
Oxygen compressor	-8.19
Nitrogen compressor	-8.21
ASU power	-23.40
Total ASU system	-39.80
Gasification	
Fans & blower	-0.28
Pumps	-0.21
Coal handling and preparation	-1.8
Ash handling	-1.5
Total	-3.8
Fuel Gas Cleaning	
Refrigeration	0.0
Compressor	-4.58
Pump	0
Net	-4.58
Syngas Cleaning	
	-0.014
Methanol Synthesis	
	0
Sulfur Recovery	
Expander	4.24
Compressor	-4.27
Pump	0.00
Net	-0.03
Total Plant	
Net plant power generation, MW	309.1
Plant net heat rate, kJ/kWh (Btu/kWh) (HHV)	10,286 (9749)
Plant net efficiency, %, (LHV)	37.77
Plant net efficiency, %, (HHV)	35.00

 Table 7.14 – Methanol Synthesis Plant Thermal Performance with Novel Gas Cleaning

Table 7.15 lists several quantities related to the use of resources and emissions in the Methanol Synthesis plant with Novel Gas Cleaning technology. Only small quantities of process condensate are generated in both cases, primarily in the Sulfur Recovery Section, and this is recycled without treatment. A moderate level of fresh process water is required in the gas cleaning processes, and both cases result in no discharge of process water. Both cases require small quantities of boiler-quality makeup water for fuel gas humidification. The total sulfur emissions are expressed in three different bases (percent removal, mass per unit fuel energy input, and mass per MWe net power generated).

Auxiliaries	
Net IP steam use, MJ/hr (10 <sup>6</sup> Btu/hr)	37,140 (35.2)
Net LP steam use, MJ/hr (10 <sup>6</sup> Btu/hr)	-18,675 (-17.7)
Total cooling water use, MJ/hr (10 <sup>6</sup> Btu/hr)	258,183 (244.7)
Total condensate generated, kg/hr (lb/hr)	5,979 (13,181)
Total process water used, kg/hr (lb/hr)	26,464 (58,343)
Net process water makeup, kg/hr (lb/hr)	20,485 (45,162)
Total boiler water makeup, kg/hr (lb/hr)	41,469 (91,421)
Emissions	
Sulfur total removal efficiency (%)	99.991
Sulfur total emission, mg/MJ (lb SO <sub>2</sub> / 10 <sup>6</sup> Btu) (HHV)	0.238 (0.000554)
Sulfur total emission, kg/MW (lb SO <sub>2</sub> / MWe)	0.00245 (0.0054)
NOx total emission, mg/MJ (lb $NO_2/10^6$ Btu) (HHV)	4.34 (0.0101)
Particulate emission, mg/MJ (lb/ 10 <sup>6</sup> Btu) (HHV)	0.0079 (1.835E-05)
Hg emission, mg/MJ (lb/TBtu) (HHV)	0.0763 (0.1774)
Feed Streams, kg/hr (lb/hr)	
Chemicals	0
Sorbents	2,070 (4,564)
Catalysts	0.9 (2)
Solid waste, kg/hr (lb/hr)	
Slag product (25 wt% water)	15,915 (35,087)
Slag & HCl sorbent waste	1,384 (3,052)
Waste salts (25 wt% water)	0
Sorbent wastes	94 (208)
Total	17,394 (38,347)

Table 7.15 – Methanol Synthesis Plant Resource Use and Emissions with Novel Gas Cleaning

### 7.3 COST ESTIMATES FOR METHANOL SYNTHESIS WITH NOVEL GAS CLEANING

No financial analysis of such a co-production plant has been performed in this evaluation. The evaluation utilizes direct comparison of gas cleaning equipment investment, annual operating cost of the gas cleaning equipment, total plant electricity production and methanol production as the basis for comparison.

The major equipment purchase costs and installed costs are listed in Table 7.16 for each of the cleaning sections of the plant evaluated. The Gasification Section's cost for the convective cooler has been included in the cost breakdown since this will differ from the convective cooler cost in the Novel Gas Cleaning process. Also, the Mercury Removal cost has been taken out of the Low-Temperature Cooling Section cost and reported as a separate item. The Low-temperature Cooling Section is the second most expensive of the gas cleaning sections, and its cost approaches the cost of the

Desulfurization Section. The total gas cleaning costs are also reported on the basis of \$ per kW of net power generated. While the total gas cleaning equipment cost approaches 100 %/kW, this is clearly only a small portion of the total plant equipment cost. The impact of gas cleaning on the overall plant performance and its operating cost are much more important factors.

Plant Section	Cost, k\$
Raw gas convective cooling	
purchased equipment	324
installed equipment	648
Gas Cleaning (fuel and syngas)	
purchased equipment	8,014
installed equipment	13,500
Mercury removal	
purchased equipment	1,435
installed equipment	2,339
Sulfur Recovery	
purchased equipment	6,192
installed equipment	10,157
Methanol Synthesis	
purchased equipment	1,517
installed equipment	2,882
Fuel gas and Purge gas expanders	
purchased equipment	1,536
installed equipment	2,304
Total	
purchased equipment	19,017
installed equipment	30,144
purchased equipment, \$/kW	57
installed equipment, \$/kW	91
Total Plant	
TCR, k\$	483,723
TCR, \$/kW	1565

Table 7.16 – Equipment Cost Breakdown - Novel Methanol Synthesis

The Total Capital Requirement for the Methanol co-production plant using conventional, lowtemperature gas cleaning technology was estimated by scaling the non-gas cleaning equipment costs from IGCC power plant cost data. A confirmed basis does not exist for making this estimate, so the total plant costs are uncertain, but represent a good basis for technology comparisons. Table 7.17 shows the estimate for the Total Capital Requirement for the plant, and lists the breakdown for the Cost-of-Electricity (COE) for the plant using by-product values for the methanol and elemental sulfur products. Coal slag is assumed to be a disposal product having no value. The objective here is not to assess the financial implications for such a plant, but it to provide a basis for comparison with the COE for a comparable plant using Novel Gas Cleaning technology.

	COE
	(Cents/kWh)
Fixed O&M	0.35
Variable O&M	0.23
Consumables	0.35
water	0.040
chemicals, sorbents, catalysts	0.189
waste Disposal	0.125
By-product credit	1.03
sulfur	0.0678
methanol	0.963
Fuel	1.81
Capital charges	3.35
Total	5.06

 Table 7.17 – Cost-of-Electricity - Novel Methanol Synthesis

Table 7.17 shows clearly that the gas cleaning process equipment cost has little impact on the COE of the plant, but the gas cleaning process influence on the plant efficiency is very important. Consumables in the conventional technology plant have a very small impact on the COE. This sensitivity perspective indicates that the gas cleaning process focus should be on minimizing performance losses rather than on minimizing equipment costs.

### 7.4 Assessment of Methanol Synthesis with Novel Gas Cleaning

### 7.4.1 Performance Potential

Table 7.18 shows that the Novel Gas Cleaning technology provides the potential for improvement in Methanol Synthesis plant water resource use and thermal performance over using conventional gas cleaning technology. The Novel Gas Cleaning technology uses less than half the cooling water rate used by the conventional gas cleaning technology. Total water consumption is about the same for both technologies. Novel Gas Cleaning technology consumes more process makeup water than the conventional gas cleaning technology, primarily for coal slurry preparation, but it uses much less boilerquality makeup water, a more expensive water source. The conventional gas cleaning technology results in a water discharge stream from the power plant, a plant export that is restricted in some locations.

The detailed power consumption breakdowns listed in this report indicate that every section of the fuel gas cleaning process, except for the sulfur recovery process, shows significant advantage for the Novel Gas Cleaning technology. Conventional fuel gas cleaning technology applies a large number of fuel gas cooling and reheating operations, conducting total condensation of the fuel gas water vapor, followed by re-humidification, and this results in significant losses in overall power plant thermal efficiency that do not occur when using the Novel Gas Cleaning technology.

	Conventional	Novel Gas
	Gas Cleaning	Cleaning
Cooling water use, MJ/hr (10 <sup>6</sup> Btu/hr)	252,169 (239)	108,675 (103)
Process water makeup, kg/hr (lb/hr)	498 (1097)	42,305 (93,266)
Boiler feed water makeup, kg/hr (lb/hr)	59,972 (132,213)	33,566 (74,000)
Total water consumption, kg/hr (lb/hr)	60,469 (133,310)	75,872 (167,266)
Net power generated (MW)	288	309
Plant net heat rate, kJ/kWh (Btu/kWh)	11,009 (10,434)	10,286 (9749)
(HHV)		
Plant net efficiency, % (LHV)	35.2	37.8
Plant net efficiency, % (HHV)	32.7	35.0

Table 7.19 lists the clean fuel gas and stack gas compositions estimated for the Methanol Synthesis. The conventional gas cleaning technology has established capability to achieve the levels of sulfur control required in the evaluation. The Novel Gas Cleaning technology is estimated as having this capability based on the development work completed. Where these gas cleaning technologies differ is that the conventional gas cleaning technology will reduce the fuel gas halide, ammonia, and HCN contents to much lower levels in the fuel gas than will the Novel Gas Cleaning technology. The Novel Gas Cleaning technology can, though, reduce the fuel gas halide, ammonia and HCN contents sufficiently to satisfy the requirements for application. The Novel Gas Cleaning technology uses partialdecomposition of ammonia in the process, and low-NOx, diffusion flame, fuel gas combustors (staged, rich-quench-lean; or catalytic) to achieve low NOx emissions from the IGCC power plant. The highertemperature operations of the Novel Gas Cleaning technology may provide advantages, with less potential for the formation of metal carbonyls, and its final stage Mercury Removal Filter-Reactor may provide additional protection against corrosion-based particulate damage to the gas turbine not provided by conventional gas cleaning technology. The conventional gas cleaning technology, with operation at conditions of high corrosion potential, is inherently more prone to availability losses than the Novel Gas Cleaning technology.

The plants produce comparable solid waste streams, with the total mass of waste for the Novel Gas Cleaning technology being about 5% greater than with the conventional gas cleaning. The nature of the waste differs slightly for the two technologies. The slag waste streams are very similar in flow rate and composition for the plants. The conventional gas cleaning technology produces a wet stream of halide salts that contain numerous traces of contaminants, as well as a small, hazardous mercury sorbent waste. The Novel Gas Cleaning technology produces dry, non-hazardous sorbent waste, some of which can be incorporated into the plant slag waste. The hazardous nature of the mercury sorbent waste from the Novel Gas Cleaning Process is uncertain at this time.

### 7.4.2 Cost Potential

The Methanol Synthesis co-production plant investment and cost-of-electricity (COE) is compared for the two plants in Table 7.20. While the equipment costs are estimated to be only slightly lower for the Novel Gas Cleaning process than for the conventional gas cleaning process, the improved power plant capacity and heat rates result in the potential for significant reductions in plant capital investment (greater than 14%) and cost of electricity (greater than 9%).

	Conventional Cleaning	Novel Gas Cleaning
Clean Fuel Gas (before N <sub>2</sub> dilution)		-
$H_2$ (vol%)	27.2	23.8
CO (vol%)	43.8	46.6
CH <sub>4</sub> (vol%)	0.3	0.3
CO <sub>2</sub> (vol%)	10.6	10.8
H <sub>2</sub> O (vol%)	16.2	19.4
N <sub>2</sub> (vol%)	1.7	7.2
Ar (vol%)	0.15	0.2
Total sulfur (ppmv)	0.24	1.9
Halides (ppbv)	0.1	3600
Ammonia (ppmv)	0.4	160
HCN (ppmv)	0	19
Hg (ppbv)	0.07	0.125
Clean Syngas		
$H_2$ (vol%)	32.4	29.1
CO (vol%)	52.3	46.8
CH <sub>4</sub> (vol%)	0.4	0.4
CO <sub>2</sub> (vol%)	12.7	13.2
$H_2O$ (vol%)	0.0	1.5
$N_2$ (vol%)	2.1	8.9
Ar (vol%)	0.18	0.24
Total sulfur (ppmv)	0.03	0.47
Halides (ppbv)	0.1	0.15
Ammonia (ppmv)	0.44	5.5
HCN (ppmv)	0.0	0.03
Hg (ppbv)	0.07	0.05
Stack Gas		
$CO_2$ (vol%)	10.1	10.0
$H_2O(vol\%)$	10.7	10.7
N <sub>2</sub> (vol%)	70.5	70.2
$O_2$ (vol%)	7.9	8.3
HCl (ppmv)	0.0	0.35
SO <sub>2</sub> (ppmv)	0.033	0.2
NO (ppmv)	<5	<5
Hg (ppbv)	0.01	0.02
Sulfur Removal		
Sulfur total removal efficiency, %	99.9985	99.9911
Sulfur total emission,	0.413 (0.00096)	2.32 (0.0054)
mg/MJ (lb SO <sub>2</sub> / $10^6$ Btu)		
Solid Waste		
Slag and flyash (wet), kg/hr (lb/hr)	16,313 (35,964)	15,915 (35,087)
Waste salts (wet), kg/hr (lb/hr)	338 (745)	0
Sorbent wastes, kg/hr (lb/hr)	1.8 (4)	1,479 (3,260)
Total, kg/hr (lb/hr)	16,653 (36,713)	17,394 (38,347)

 Table 7.19 – Methanol Synthesis Gas Cleaning Emission Comparisons

Gas Cleaning Technology	Conventional Gas Cleaning	Novel Gas Cleaning
Generation capacity, MWe	288	309
Plant Heat Rate, kJ/kWh (Btu/kWh) (HHV)	11,009 (10,434)	10,286 (9749)
Total Capital Requirement, \$/kW	1791	1565
Total COE, cents/kWh (constant \$)	5.6	5.1

 Table 7.20 - Methanol Synthesis Plant Investment and COE Comparison

### 8. CONCLUSIONS AND RECOMMENDATIONS

This evaluation has devised plausible humid-gas cleaning schemes for the Filter-Reactor Novel Gas Cleaning process that might be applied in IGCC and Methanol Synthesis applications. These schemes are simpler than those used in conventional dry-gas cleaning for these applications and show the conceptual-potential to provide plant availability, plant thermal efficiency and cost improvements over the conventional plants.

The Filter-Reactor should have a basic design similar to the design of near-commercial barrier filters, with a large number of independently pulse-cleaned filter plenums that allow the Filter-Reactor to maintain high levels of emission control. Sorbent particle sizes injected into the Filter-Reactors are expected to operate best at -325 mesh, with a mass-mean size of about 20 µm. The major uncertainties have been 1) the contaminant removal performance that can actually be achieved in these Filter-Reactors, with their relatively thin 5 to 13 mm (0.2 to 0.5 inch) sorbent filter cakes and low gas velocities through the filter cakes, and 2) the possible reaction-sintering behavior of the filter cakes that might occur at the stage conditions. These uncertainties have been resolved in the program's PDU tests under representative conditions.

Detailed material & energy balances for the gas cleaning applications, coupled with preliminary thermodynamic modeling and laboratory testing of candidate sorbents, have identified the probable sorbent types that should be used, their needed operating conditions in each stage, and their required levels of performance. These performance goals and the results from the PDU testing are summarized in Table 1.4. In general, the performance goals have been demonstrated in the PDU testing, with the exceptions noted in the table. A water scrubbing stage is used for syngas polishing of halides and ammonia in the Methanol Synthesis application, and this stage should be able to be applied commercial using available technology experience. The conditions and performance levels that have not been demonstrated in the PDU testing in this program have been extrapolated from the PDU test results to apply to the commercial design and evaluations.

The evaluation utilized a regenerative, zinc-based sulfur sorbent in a transport reactor configuration for bulk sulfur removal, but the Filter-Reactor Novel Gas Cleaning process can be coupled with any developing bulk desulfurization technology (such as alternative sorbents and alternative gassorbent bulk desulfurization contactors) operating under humid-gas conditions. The use of alternative bulk desulfurization technology will alter some of the Filter-Reactor stage conditions and sorbents.

The success of PDU tests completed in the program, and the conceptual advantages of the Filter-Reactor technology indicated by the evaluation results lead to conclusion that continued scale-up development of the technology is merited. This development should focus on the optimization of the Filter-Reactor performance (operating face velocity, sorbent feed rate, sorbent properties and size distribution, operating temperature, simultaneous removal of multiple contaminants), Filter-Reactor scaleup through larger-scale test units, and continued commercial process evaluation.

Cleaning Stage	Sorbent type (-325 mesh)	Process Temperature °C (°F)	Process Performance Goals	PDU Test Status (Final Report Volume II)
IGCC App	lications			
Bulk halide removal	Sodium mineral (Trona or Nahcolite)	593 (1100)	<ul><li>99% halide removal,</li><li>5 ppmv HCl outlet,</li><li>Na/Cl mole feed ratio 4,</li><li>75% ammonia decomposition.</li></ul>	Demonstrated at 427°C (800°F), Ammonia decomposition not measured (not in program scope)
Sulfur polishing	Zinc-titanate	482 (900)	96% removal, 40 ppmv inlet to 2 ppmv outlet, Zn/S mole feed ratio 3.	Not considered in PDU tests (focus placed on more challenging Methanol sulfur polishing)
Mercury removal	TDA sorbent	204-316 (400-600)	90-95% Hg removal, Sorbent/Hg mass feed ratio 1000, Possibly simultaneous with sulfur polishing.	90% Hg removal demonstrated at 260°C (500°F), Simultaneous sulfur removal not attempted (insufficient test time).
Methanol S	ynthesis Applic	ation		
Halide & ammonia polishing	Water absorbent	93-149 inlet (200-300)	97% ammonia removal to 10 ppmv, 99.8% HCl removal to 10 ppbv.	Halide and ammonia scrubbing not addressed in PDU tests (design from scrubbing experience).
Sulfur polishing	Zinc-titanate	260-316 (500-600)	98% sulfur removal, 60 ppbv sulfur outlet, Zn/S mole ratio 5.	Sulfur polishing demonstrated in PDU tests.
Mercury removal	TDA sorbent	204-316 (400-600)	95% Hg removal, Sorbent/Hg mass feed ratio 1000.	90% Hg removal demonstrated at 260°C (500°F) (data extrapolated for design).

## Table 8.1 – Filter-Reactor Novel Gas Cleaning Stage Performance Goals and Test Status

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# NOVEL GAS CLEANING/ CONDITIONING FOR INTEGRATED GASIFICATION COMBINED CYCLE

# **VOLUME II – PROCESS DEVELOPMENT UNIT (PDU) TEST RESULTS**

## **OPTIONAL PROGRAM FINAL REPORT**

September 1, 2001 - December 31, 2005

By

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# ACRONYMS AND ABBREVIATIONS

BCB	Bethlehem Coke Breeze
Btu	British Thermal Unit
CF-R	Conditioning Filter-Reactor
CRS	GTI's Chemical Research Services Department
DOE	Department of Energy
DSQ	Direct Spray Quench
EPA	Environmental Protection Agency
FFTF	Flex-Fuel Test Facility
FPD	Flame Photometric Detector
FT-IR	Fourier Transform Infrared Spectrometer
GC	Gas Chromatograph
GRE/CCS	Great River Energy Coal Creek Station
GRI	Gas Research Institute
GRI	Gas Research Institute
GTI	Gas Technology Institute
IC	Ion Chromatograph
IGCC	Integrated Gasification Combined Cycle
IMACC	Industrial Machine and Control Corporation
JM	Johnson Matthey
MAF	Moisture and Ash Free
MS	Mass Spectrometer
MW	Molecular Weight
NDIR	Non-Dispersive Infrared
NETL	National Energy Technology Laboratory
NGC	Novel Gas Cleaning
NGC-OPT1	Novel Gas Cleaning Optional Program Test Campaign #1
NGC-OPT2	Novel Gas Cleaning Optional Program Test Campaign #2
NGC-OPT3	Novel Gas Cleaning Optional Program Test Campaign #3
P&ID	Piping and Instrumentation Diagram
PDU	Process Demonstration Unit
PFD	Process Flow Diagram
PFPD	Pulsed Flame Photometric Detector
ppbv	Parts per billion by volume
ppmv	Parts per million by volume
ppmw	Parts per million by weight
PRDA	Program Research and Development Announcement
SCF	Standard Cubic Feet
SE&C	Sample Extraction and Conditioning
SGB	Sulfur Guard Bed
Siemens	Siemens Power Generation, Inc.
TC	Trim Cooler
TCD	Thermal Conductivity Detector
TDA TE D	TDA Research, Inc.
TF-R	Test Filter-Reactor
UAB	University of Alabama at Birmingham

## **1. EXECUTIVE SUMMARY**

Much of the hot gas cleanup research and development efforts have been conducted within the context of the Integrated Gasification Combined Cycle (IGCC), focusing predominantly on the development of regenerable metal oxide sorbents for the removal of reduced sulfur compounds (mainly hydrogen sulfide (H<sub>2</sub>S) and carbonyl sulfide (COS)) at high temperature and pressure. The objective in this application is to reduce the H<sub>2</sub>S concentration in the fuel gas to less than 20 ppmv to satisfy gas turbine requirements. In recent years there has been significant interest in extending the hot gas cleanup capabilities so that the cleaned fuel gas becomes suitable for a wide range of applications, including fuel cells and production of chemicals and transportation fuels, in addition to electric power generation via the IGCC technology. These applications require the removal to near-zero levels, of the sulfur-, nitrogen-, alkali-, and chlorine-containing gas emissions and fine particulate matter (PM<sub>2.5</sub>) caused by fuel bound constituents that are naturally present in many forms in a variety of carbonaceous materials which are useful as feedstocks to gasification processes. When the IGCC system is applied to creating synthesis gas for production of liquid products (e.g., methanol, F-T liquids), these species can be poisonous to downstream processing units and may be contaminants in the high purity products. HCl in particular is a significant contaminant that causes severe deterioration to various fuel cell materials.

Commercial technology is available to clean coal-gasification hydrocarbon gases to the stringent levels required for these demanding applications. The prevalent commercial gas cleaning process, capable of achieving the very stringent gas cleaning requirements for these types of applications, is based on the "Rectisol" gas desulfurization technology. The Rectisol process uses refrigerated methanol absorption of sulfur species and is expensive to build and operate, consuming extensive power. Commercial gas cleaning processes also apply wet, low-temperature removal of halides, particulates, and other contaminants, resulting in extensive water treatment requirements. New, cheaper technologies are needed for stringent gas cleaning duty if coal is to become competitive with cleaner fuel sources, such as natural gas, in U.S. Department of Energy advanced, near-zero emission, efficient multi-production energy plants.

To address this need, the Siemens Power Generation, Inc. (Siemens) and the Gas Technology Institute (GTI) have been developing a novel process compatible with known gasifier systems that promises, in a pre-combustion treatment, to prepare carbonaceous feedstock-based fuel gases or syngases for use in power generation applications with gas turbines or with fuel cells, or for co-production of power with chemicals or transportation fuels. This Novel Gas Cleaning (NGC) Process can be applied as an add-on cleaning step following a near-commercial, state-of-the-art hot gas polishing system. The process can also be a stand-alone fuel gas cleanup system, depending on the carbonaceous fuel properties, the extent of in-gasifier cleaning (bulk sulfur removal), and the cleanup requirements of the intended application. This novel process specifically addresses sulfur, chloride, mercury, and particulate removal to meet the most stringent requirements (total sulfur (mainly H<sub>2</sub>S and COS) < 60 ppbv, halide (mainly HCl) < 10 ppbv, Hg < 0.01 ppbv, and particulate < 0.1 ppmw). These are contaminant levels below detection limits of conventional measurement instrumentation. In addition, various options have been conceptualized to incorporate the removal of other contaminants, such as ammonia (NH<sub>3</sub>), into the NGC Process.

### **Novel Gas Cleaning Process**

A schematic flow diagram of the NGC Process configuration, for simultaneous control of particulate matter and sulfur and halide gaseous contaminants, is shown in Figure 1. It is a dry process that follows a hot, or warm, bulk desulfurization step. The process injects fine sulfur and halide sorbent particles into two stages of barrier filter-reactors integrated in series, coupling efficient particle capture with an effective entrained and filter cake reaction environment. The first stage injects inexpensive, fine sodium-based and zinc-based sorbents into the hot (371 to 593°C; 700 to 1100°F) fuel gas stream for

reducing the concentration of the primary contaminants ( $H_2S$  and HCl) to about the 1-ppmv level. In the second stage, the fuel gas exiting from the first barrier filter-reactor is first cooled to 260 to 316°C (500 to 600°F), and then injected with highly reactive, fine particles of ZnO-containing materials for sulfur removal and Na<sub>2</sub>CO<sub>3</sub>- or Na<sub>2</sub>O-containing materials for halide removal before entering a second barrier filter-reactor, where contaminant concentrations are further reduced to the required levels.

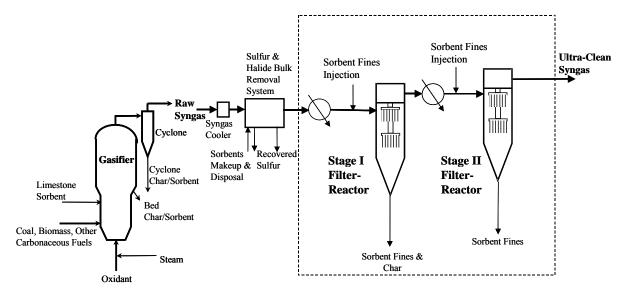


Figure 1 - Schematic Diagram of the Novel Gas Cleaning Process

The main basis for the NGC Process concept is that, in addition to filtration, barrier filters provide ideal environments for chemical reactions. Syngas contaminant (e.g., HCl, H<sub>2</sub>S, COS, etc.) removal via fine sorbent particles injected into the syngas upstream of the barrier filter occurs both from the dilute, entrained sorbent particles and within the consolidated filter cake. The nature of the filter cake is modified by the low ash content of the fuel gas and the relatively high content of injected sorbent particles. The high permeability of the resulting filter cake, compared to the normally very low permeability of gasifier ashes, allows for higher face velocity design, resulting in a more compact vessel having lower cost than the typical fuel gas hot gas filter. Only a portion of the filter elements (about 5%) are pulse cleaned at any given time, so the loss in the contaminant removal performance of the barrier filter-reactor due to loss of filter cake contaminant removal capacity will be very small.

As described in Volume I the NGC Process concept has many merits and provides for a "onebox" solution to hot/warm syngas cleaning. For example, there is significant potential to incorporate, into the NGC Process, the capability to control mercury emissions to meet syngas mercury removal requirements for chemical synthesis applications (as low as 0.01 ppbv in the syngas, or > 99% removal) and for IGCC applications (< 0.45 kg of mercury per 1.06 trillion kJ (< 1 lb of mercury per trillion Btu), or about 90% removal). Mercury control can be accomplished through injection of an appropriate sorbent into the second stage of the NGC Process, or in a dedicated downstream packed-bed reactor operating at a lower temperature.

The overall Novel Gas Cleaning Process development program consisted of two phases: a Base Program phase and an Optional Program phase. The goal of the Base Program (completed in August 2001) was to provide the necessary data to verify the technical and economic feasibility of the NGC Process. GTI's scope of work in this first phase was dedicated most heavily to the laboratory testing of selected candidate sorbent materials for removal of reduced sulfur species (primarily H<sub>2</sub>S and COS) and halide species (primarily HCl) from simulated fuel gas mixtures. Experimental approaches were designed

to closely simulate the gas/solid contact as well as the operating conditions in the NGC Process. Analytical techniques were developed for the measurement of sulfur and chloride species at trace levels to generate reaction conversion performance data. The most suitable desulfurization and dechlorination materials were identified and the optimum ranges of operating conditions determined for each stage of the NGC Process to achieve the target contaminant levels ( $H_2S < 60$  ppbv and HCl < 10 ppbv) in the cleaned fuel gas with acceptable conversion rates (i.e., sorbent feed rates).

The Base Program provided for laboratory and conceptual exploration of the concept and derived an indication of its feasibility, leading to selection of the concept for further support. The second phase of the NGC Process development program (Optional Program) was exercised for conducting pilot-scale experiments and performing thorough economic, technical, and commercial feasibility analyses. The main goal of the Optional Program was to conduct a proof-of-principles demonstration of the integrated NGC Process on a GTI carbonaceous fuel gasifier producing raw fuel gases representative of commercial gasifier practice, to confirm technical and economic feasibility of the process. An important objective of the Optional Program was to verify, at the pilot-scale, the laboratory test results obtained in the Base Program with respect to operating temperatures for Stages I and II of the NGC Process, sorbent selections, sorbent/contaminant ratios, and ability of the process to accomplish very stringent contaminant levels in the syngas (i.e., particulates < 0.1 ppmw, total reduced sulfur compounds (H<sub>2</sub>S, COS) < 60 ppby, and total halides (mainly HCl) < 10 ppby). In addition to particulates and sulfur and halide species, efforts were devoted in the Optional Program to evaluate the potential of incorporating mercury control into the NGC Process, with the objective of reducing mercury (mainly elemental mercury, Hg<sup>0</sup>) concentration in the syngas to less than 0.01 ppby, corresponding to mercury removal efficiencies > 99%.

### **Preparation of Pilot-Scale Facilities**

The original plan was to refurbish an existing GTI gasifier to generate syngas via coal gasification for the NGC Process testing. Project schedule, however, coincided with GTI's decision to construct a new carbonaceous fuel gasification facility (Flex-Fuel Test Facility) with capability to process about 10 tons of coal per day. The overall Flex-Fuel Test Facility configuration includes the fluidized-bed gasifier, the NGC Process section, and the exhaust gas handling and cleanup section. Preparation of the NGC Process section was conducted in parallel with the Flex-Fuel Test Facility design and construction activities. This included designing a suitable process configuration and specifying, procuring, and installing the necessary pieces of equipment. The final NGC Process section layout, shown schematically in Figure 2, was determined after evaluation of several options and redirection of the program goals to focus on the more challenging aspects of the project, i.e., to achieve very stringent contaminant removal targets at the ppby level. This part of the plant consists of two major sections: Conditioning and Testing. In the Conditioning section, the raw gasifier gas is treated to meet the requirements of the NGC Process Testing section (i.e., Stage II). The Conditioning section, however, did incorporate one of Siemens' barrier filters primarily to control syngas particulates, but also to remove the bulk of halides (i.e., HCl). Therefore, this first barrier-filter reactor represented Stage I in the NGC Process base configuration (i.e., Figure 1).

As shown in Figure 2, the NGC Process section consists of several major components:

## **Conditioning Section**

- Direct Spray Water Quench (DSQ)
- Conditioning Filter-Reactor (CF-R)
- Conditioning halide sorbent feed system (Stage I halide sorbent)
- Conditioning char and spent sorbent handling system
- Pressure letdown valve
- Sulfur Guard Bed and associated bypass line and pre-heater
- Syngas Trim Cooler

#### Test Section

- Test Filter-Reactor, TF-R (for Stage II testing)
- Test sulfur, halide, and mercury sorbent feed systems (for Stage II testing)
- Test spent sorbent handling system

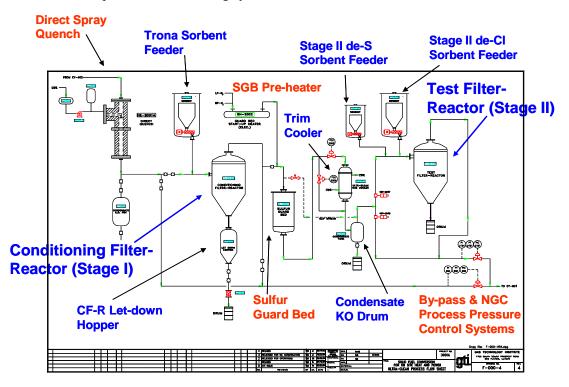


Figure 2 - P&ID for the Ultra-Clean Process Section in the Flex-Fuel Test Facility

To accomplish the NGC Process PDU test program goals, GTI, in collaboration with Siemens, developed a sampling and analysis protocol, as part of the Test Plan for the program, which defined the sampling locations, sample type (solid, gas, or liquid), sample description, purpose, number of samples, analyses to be performed, and measurement methods, during each PDU test campaign. Every effort was made to ensure that the state of the raw syngas was well defined before and after conditioning, and most importantly that performance of Stage II of the NGC Process could be properly assessed. Moreover, sufficient data would be collected to develop adequate material and energy balances for the gasifier to assess its efficiency in generating syngas from the test feedstock. The data generated would also permit characterization of process emissions for permitting activities and gasification and syngas cleanup byproduct materials for assessment of disposal options. Process samples included fuel feedstock, ash, raw syngas, fresh sorbent materials, spent sorbents, and condensed liquids, in addition to several gas samples throughout the NGC Process section.

To meet the very stringent requirements of the analytical work scope in the NGC Process PDU test program, one double-train and two single-train sample extraction and conditioning (SE&C) skids were designed and installed at three main sampling locations: Conditioning Filter-Reactor inlet (SE&C Skid #1), Conditioning Filter-Reactor outlet (SE&C Skid #2), and Test Filter-Reactor outlet (SE&C Skid #3). These three SE&C systems use the controlled condensation management approach, which incorporates filtration, gas cooling, condensate collection and removal, and drying. This process interface approach provides for separation of suspended particles, condensates, and gases. Particles and condensates are accumulated in the skid for post-test recovery and analysis. The gases, before and after drying stages, can be collected in sample canisters, passed through collection impingers (i.e., for HCl

sampling) or absorption traps (i.e., carbon traps for mercury sampling, for example), or sent directly to analytical instruments for on-line measurements.

Two analytical stations were set up in the FFTF. The first station included four instruments: an Industrial Machine and Control Corporation (IMACC) Fourier-Transform Infrared Spectrometer (FT-IR), a Stanford Research Systems QMS300 Mass Spectrometer (MS), an Agilent 5890 gas chromatograph with flame photometric detector (GC/FPD), and a Varian micro gas chromatograph with thermal conductivity detector ( $\mu$ GC). The gas sampling system at this station was built to allow for the selection of samples from any of three sources from the process (i.e., CF-R inlet, CF-R outlet, and conditioned syngas) and direct the gas stream to one of the above four analyzers. In addition to supplying the on-line analyzers with representative syngas samples, the sampling system was used to extract sample gases to EPA Method 26 impingers, allowing for direct comparison of batch and FT-IR HCl measurements.

The second analytical station was set up in order to meet the analytical work scope requirements of the second test campaign during which critical measurements were made to assess the NGC Process Stage II performance for removal of multiple contaminants (H<sub>2</sub>S, COS, HCl) to ppbv levels, and those of the third test campaign during which mercury removal via dry sorbent injection was investigated. This station included a Varian gas chromatograph featuring a built-in high performance sample concentrator, a sensitive pulsed flame photometric detector (PFPD) with a detection limit of 30 to 50 parts-per-trillion (ppt), and significant capability for speciated and total sulfur analyses. For on-line Hg analysis, a Tekran Mercury Vapour Analyzer 2537A was set up at the TF-R outlet that was much more sensitive than the off-line instrument, a Nippon WA-4 Mercury Analyzer (~ 0.01  $\mu$ g/m<sup>3</sup> detection limit). To measure HCl at the ppmv and ppbv levels, glass impingers were setup at both analytical stations for chlorine sampling according to EPA Method 26, for subsequent analysis using an ion chromatograph instrument (Dionex ICS-1000).

The sampling systems were installed to measure reduced sulfur (H<sub>2</sub>S and COS) and halide (HCl) compounds at various concentrations throughout the NGC Process section, ranging from hundreds of parts-per-million (ppmv) to very low concentrations at the parts-per-billion (ppbv) level. Extreme care was taken in the selection of construction materials for sampling to provide very low detection and to avoid contamination and loss of analyte. All sample transport lines from the various process interface points to the analytical stations were heat-traced and insulated to preserve gas sample integrity and to maintain gas temperatures above the instrument inlet conditions. Components of the SE&C skids at the G-13 and G-19 locations (tubing, fittings, valves, flow metering orifices, and Mott filters) and sample transport lines were coated with chemically-inert Silcosteel<sup>®</sup> and Sulfinert<sup>®</sup> coatings to minimize reactions and adsorption of gases on the surfaces. Sample canisters and valves were made from Sulfinert<sup>®</sup> materials. Low pressure and non-heated sampling lines and vessels were constructed of Teflon or borosilicate glass. Flexible silicone tubing was used to make low pressure connections between the coated steel or Teflon tubing and glass apparatus. The sampling methodology was validated throughout testing by purging sampling points with dry N<sub>2</sub> and reference gases containing certified concentrations of HCl and H<sub>2</sub>S.

#### **PDU Test Materials and Scope of Work**

The pilot-scale test program comprised three one-month test campaigns. Table 1 lists the sorbents and operating conditions as well as the estimated, nominal stream flows and pressures for the Conditioning Filter-Reactor and the Test Filter-Reactor that would be applied in these three test campaigns. All of the sorbents are commercial, with the possible exception of the two mercury sorbents, which had been selected through a comprehensive laboratory test program and interactions with other R&D organizations. The Stage I and Stage II inlet temperatures are controlled process variables in the PDU test program. The syngas pressure is fixed in a given test campaign, and the syngas flow rate is set by the Flex-Fuel gasifier operating capacity. The inlet sulfur and halide contaminant levels are also controlled process variables, but with some uncertainty as to the levels of control that can be achieved in

the process. The ranges of sorbent feed rates listed (moles of sorbent active component divided by the moles of the specific contaminant) reflect the expected range that might be required to achieve the targeted gas cleaning performance.

Pilot-scale testing materials stocks were determined based on estimates of NGC Process flows, test campaign durations, and other considerations. Test materials included a relatively low-sulfur carbonaceous feedstock (metallurgical coke (0.69% S, 800 ppmw Cl, 0.02 ppmw Hg), North Dakota Lignite (0.89% S, ~ 10 ppmw Cl, 0.06 ppmw Hg), and a high-ash coal from India (0.56% S, < 100 ppmw Cl, 0.14 ppmw Hg) that was available from a recent GTI project), trona (and nahcolite as an alternative material) for bulk HCl removal in the Conditioning Filter-Reactor, BASF's R5-12 catalyst or Süd - Chemie's G-72E catalyst as a ZnO sulfur guard bed material, Süd -Chemie's G-72E and G-92C catalysts as Stage II desulfurization and dechlorination sorbents, respectively. Except for trona and nahcolite, which were procured in fine form from a commercial supplier, all of the sorbents are sized (ground and sieved) to -325 mesh at GTI, with an estimated mass-mean diameter of 20 μm.

Filter-Reactor	Conditioning	Test
Inlet temperature (°F)	830	550
Inlet pressure (psia)	277	215
Inlet syngas flow (lb/hr)	3439	3648
Filter Face velocity (ft/min)	2.7	3.1
Inlet total sulfur content (ppmv)	not applicable	1 – 5

150 - 500

None

Trona

not applicable

1 - 5

2 - 10

None

None

1 - 5

G-72E

G-92C

0.06

0.01

2 - 10

GTI-Hg-S9 TDA's Hg Removal Sorbent 1000 - 2000

 Table 1 - Conditioning Filter-Reactor and Test Filter-Reactor Nominal Test Conditions

#### **Results and Discussion**

Inlet halide content (ppmv)

Sulfur sorbent (-325 mesh)

Halide sorbent (-325 mesh)

(mole/mole contaminant) Mercury sorbents (-325 mesh)

Mercury sorbent feed rate

(sorbent-to-Hg<sup>0</sup> mass ratio)

Target sulfur outlet content (ppmv)

Target halide outlet content (ppmv)

Sulfur and halide sorbent feed rates

#### NGC Test Campaign One (NGC-OPT1)

The primary objective of the Novel Gas Cleaning Optional Program Test Campaign 1 (NGC-OPT1) was to evaluate the performance of the Conditioning Filter-Reactor for combined particulate/halide removal from syngas derived from the gasification of a carbonaceous feedstock. This first test campaign was conducted primarily with metallurgical coke (Bethlehem Coke Breeze), whose chloride content (~ 800 ppmw) best suited the test objectives. In addition to the Flex-Fuel gasifier, the Direct Spray Quench system and the Conditioning Filter-Reactor and associated halide sorbent (trona) injection system and pulse control skid were successfully commissioned and tested during this test campaign.

Gasification of Bethlehem Coke Breeze and washed Indian coal was conducted successfully for  $\sim$  62 hours. This campaign comprised four test segments with three representing steady-state testing periods involving operation with metallurgical coke without trona injection, metallurgical coke with trona injection, and Indian coal without trona injection. Trona was injected into the syngas immediately

upstream of the CF-R vessel for  $\sim$  18 hours. Pulsing cycles during operation with metallurgical coke were long (once every 2 to 4 hours), but increased to approximately 7-8 pulses per hour as the solids loading in the syngas increased significantly during operation with the high-ash Indian coal. In both cases, however, the filter pressure drop behavior was stable.

Using certified gas mixtures containing appropriate HCl and  $H_2S$  concentrations and spike-andrecovery techniques, the high-temperature process interface approach for syngas sample extraction and conditioning was validated at the CF-R inlet and outlet locations. Reproducibility of the impinger sampling/ion chromatograph chloride measurements was also demonstrated. The EPA Method 26 impinger/ion chromatograph batch HCl sampling and measurements and on-line FT-IR HCl analyses showed very good agreement. Both approaches were able to identify and quantify the effect of char on HCl concentrations in the raw syngas, leading to confirmation of the expected HCl concentration (106 ppmv was measured compared to an expected HCl concentration of ~ 111 ppmv).

Both time-averaged (batch) impinger measurements and on-line FT-IR analyses indicated that the HCl concentration in the CF-R product syngas slowly decreased as the trona injection rate was increased. The lowest HCl concentration in the CF-R outlet syngas was measured at 10.8 ppmv during trona injection, corresponding to ~ 90% HCl removal in the CF-R vessel. This lower than expected HCl removal performance could be due to several factors, including lower reaction temperature, high raw syngas steam content (equilibrium limitation), higher size distribution of the injected trona, lower face velocity (syngas flow rate). The influence of each of these parameters was examined and it appears likely that a large portion of the trona fed would not have deposited on the filter elements given the relatively large portion of large particles in the trona, and the low syngas velocity through the CF-R vessel. Because metallurgical coke was used in this test campaign, the syngas flow rate was limited to ~ 1800 lbs/hr, which corresponds to a face velocity of  $\sim 2$  ft/min (a face velocity > 3 ft/min is desired). In addition, posttest analyses indicated the particle size distribution of the as-received Solvay T-200® trona was much coarser than the char. The volume moment mean diameter (i.e., mean diameter with respect to the mass of the particles) is about 13  $\mu$ m and the median diameter, d(0.5), is about 8  $\mu$ m for the char sample. The corresponding values for the bulk dechlorination material were significantly higher, 53.8 µm and 35.6 μm, respectively.

#### NGC Test Campaign Two (NGC-OPT2)

The primary objective of the NGC-OPT2 was to evaluate the performance of the Test Filter-Reactor (TF-R) for sulfur (H<sub>2</sub>S and COS) and halide (HCl) removal (individually and simultaneously) to very stringent levels from syngas derived from the gasification of a carbonaceous feedstock. The washed Indian coal was selected as the primary feedstock for this test campaign, due to its chloride and sulfur content. After the test objectives were achieved, the coal feed was switched to North Dakota lignite to evaluate this fuel's suitability for the third test campaign focusing on mercury control. The entire Flex-Fuel Test Facility (gasification and NGC Process sections) was successfully operated throughout the NGC-OPT2A (week of March 21) and the NGC-OPT2B (week of March 28) test campaigns, including two Siemens barrier filters (CF-R and TF-R), Sulfur Guard Bed (SGB) and associated low-pressure nitrogen pre-heater and slipstream by-pass system, and Stage II sulfur (G-72E) and halide (G-92C) sorbent feeders. The low HCl content of the raw syngas (10 to 20 ppmv) made it possible to forego trona injection into the syngas upstream of Stage I and focus solely on the more challenging aspects of the process, i.e., achieving parts-per-billion (ppbv) levels in the ultra-cleaned Stage II product syngas. The Trim Cooler (located between the SGB and TF-R) was not utilized in these tests. In addition, G-92C halide sorbent was fed as a 50/50 physical mixture with nahcolite to improve its feeding as determined in prior commissioning tests.

The raw syngas, derived from the gasification of primarily washed Indian coal, was successfully conditioned to the temperatures and contaminant levels required at the inlet to the TF-R. Throughout testing, temperatures across the NGC Process section (Figure 2) were maintained at  $\sim 1450^{\circ}$ F at the

secondary cyclone, ~ 1400°F at the inlet to the Direct Spray Quench, ~ 700°F at the inlet to the CF-R, ~ 650°F at the outlet of the CF-R, ~ 650°F at the SGB inlet, ~ 600°F at the SGB outlet, ~ 600°F at the TF-R inlet, and ~ 550°F at the TF-R outlet. These conditions ensured operation of the SGB at optimal conditions for maximized utilization of the SGB catalyst and efficient sulfur removal. These conditions also ensured that contaminant removal reactions across the TF-R vessel were kept within the optimum temperature range (550°F to 575°F). The total sulfur concentration (H<sub>2</sub>S and COS) at the inlet was regulated within a 1-5 ppmv range throughout testing by maintaining a regulated syngas slipstream that bypassed the SGB vessel. An on-line GC/FPD provided near-continuous H<sub>2</sub>S and COS measurements. In addition, because of low levels of HCl in the raw syngas (about 20 ppmv) and some measured HCl removal in the CF-R vessel (through interaction with char and residual trona) and in the SGB vessel (a small amount of CaO in the SGB catalyst), it was not necessary to engage the trona sorbent feeder upstream of the CF-R for bulk HCl removal. The measured HCl concentration in the syngas at the G-14 location (TF-R inlet) consistently averaged ~ 2 ppmv. HCl measurements were made using both the on-line IMACC FT-IR instrument as well as the batch impinger sampling with ion chromatography analysis.

The second test campaign comprised several test segments, including halide removal by injection of finely-ground G-92C sorbent, sulfur removal via injection of the finely ground G-72E sorbent, combined removal of HCl and sulfur species (H<sub>2</sub>S and COS) via simultaneous injection of both sorbents from two separate feeders (Figure 2), and other segments of interest. The gasification section was operated under steady state conditions throughout these test segments as demonstrated by stable gasifier output. Conditioning the syngas (at the TF-R inlet) and halide removal by injection of ground G-92C sorbent were the focus of the initial phase of the second test campaign. The halide sorbent feeder functioned very well with a 50/50 physical mixture of G-92C and nahcolite which was found to facilitate feeding in prior commissioning tests. During this week of testing, efforts concentrated on controlling HCl in the TF-R and monitoring sulfur species concentrations. It was clearly demonstrated that the injected material reached the filter elements (candles):  $\Delta P$  across the filter increased steadily reaching ~ 150 in. H<sub>2</sub>O by the time this test phase was completed. Initially, the G-92C/nahcolite mixture was injected at a higher rate than was necessary (~ 100 g/min) to "pre-coat" the candle surfaces with sorbent materials and expedite testing. Within 2.5 hours,  $\Delta P$  across the TF-R reached ~ 75 in. H<sub>2</sub>O. Sorbent injection was then continued at the test design rate of 20 g/min. The Test Filter-Reactor was not pulsed during testing.

Throughout testing, numerous samples were taken from a variety of locations and were analyzed using a wide array of instrumental methods. During the second test campaign approximately 55 impinger/ion chromatography analyses and 78 gas sample canisters involving the main four sampling points as well as validation runs were taken, in addition to data taken with online instruments. During halide sorbent injection, HCl concentrations were measured to be about 58, 61, and 52 ppbv. Each of these concentrations was measured from batch impinger samples that were obtained over a 2-hour sampling period. Taking into account the background HCl concentration that was estimated to range from 40 to 55 ppbv based on blank runs, these measurements would represent approximately 3-18, 6-21, and 0-12 ppbv HCl in the ultra-clean syngas.

At the conclusion of the HCl removal testing the Test Filter-Reactor was pulsed and the sulfur removal test segment was started with pre-coating the filter elements (candles) by feeding the finely-ground G-72E sorbent at the higher rate of 30 g/min, and when the filter  $\Delta P$  reached ~ 75 in. H<sub>2</sub>O, the feed rate was reduced to 5 g/min. As in the previous halide sorbent injection test completed in the prior week,  $\Delta P$  across the second filter increased steadily. The total sulfur concentration (H<sub>2</sub>S and COS) in the TF-R outlet was measured consistently at less than 50 ppbv, using batch samples. Semi-continuous online sulfur measurements (every 24 minutes) using the Varian CP-3800 PFPD gas chromatograph were quite stable, averaging ~ 20 ppbv, much lower than the 60 ppbv target. During this test segment the total sulfur (H<sub>2</sub>S + COS) level was maintained in the 2-5 ppmv range at the TF-R inlet through increasing the amount of raw syngas that was bypassed around the SGB vessel.

Following the sulfur removal test segment, the Test Filter-Reactor was pulsed and readied for the next test segment: the combined removal of sulfur and halide species (H<sub>2</sub>S, COS, HCl). A "pre-coating phase" was first performed with the G-92C/nahcolite mixture at 50 g/min and the G-72E sorbent at 20 g/min. Within an hour,  $\Delta P$  across the TF-R increased to ~ 95 in. H<sub>2</sub>O. Combined contaminant removal continued for approximately 5 hours, when  $\Delta P$  across the TF-R reached 150 in. H<sub>2</sub>O. However, instead of pulsing the TF-R, it was decided to stop sorbent injection and continue to measure contaminant concentrations at the inlet and outlet of the TF-R for an extended period of time. Furthermore, to develop additional insights into the nature, amount, and uniformity of the filter cake on the TF-R candles, the total sulfur concentration at the TF-R inlet was raised to ~ 25 ppmv. The total sulfur concentration in the TF-R outlet was monitored overnight. Throughout this period, the total sulfur concentration ranged between 20 and 40 ppby, indicating the efficacy of the Test Filter-Reactor in removing sulfur and the NGC Process flexibility with respect to regulating the sulfur level at the Stage II inlet. Thus, it appears that sulfur levels at the TF-R inlet do not need to be maintained within a strict 1-5 ppmv range to achieve a desired output sulfur level of < 60 ppby. Successful simultaneous removal of sulfur and halide species to ultra-clean levels was also demonstrated with syngas generated by gasifying lignite. The same cleaning efficiencies during this test (with lignite) were measured as were achieved with the washed Indian coal, showing that the NGC Process properly functions with two diverse fuels.

#### NGC Test Campaign Three (NGC-OPT3)

The primary objective of the third test campaign (NGC-OPT3) was to evaluate the performance of the Test Filter-Reactor for mercury removal to very stringent levels (> 90%) from syngas derived from the gasification of a carbonaceous feedstock. North Dakota lignite was used exclusively in this campaign given its suitable sulfur, chloride, and mercury content. Selection and laboratory evaluation of candidate mercury-removal sorbents had been completed earlier in the project, identifying two candidate sorbents with potential to capture mercury to levels below 0.01 ppb in the NGC Process configuration. During selected test periods, Stage II sulfur and halide sorbents were injected, simultaneously with the Hg sorbent, into the conditioned syngas upstream of the TF-R to assess their effects on the Hg sorbent performance. GTI demonstrated the capability to measure mercury at ppb levels in coal-derived syngas and obtain reasonable material balance. The results obtained also established evidence of a significant level of mercury capture (50-75%) with one of the two selected sorbents at the relatively high temperature of 572°F (optimum NGC Process Stage II operating temperature for sulfur and halide removal). Additional testing is needed to evaluate mercury removal at lower temperatures (350 to 450°F) using both selected sorbents.

#### **Conclusions and Recommendations**

Three test campaigns were successfully completed as proof-of-principle demonstration of the NGC Process "filter-reactor" concept configuration in full integration with GTI's Flex-Fuel Test Facility. Extensive efforts in these tests were devoted to designing, installing, and validating state-of-the-art gas sampling equipment and instruments to meet the very stringent analytical needs of the program, involving measurements of concentrations of various compounds ranging from hundreds of parts-per-million (ppmv) to very low concentrations at the parts-per-billion (ppbv) level. The test program clearly demonstrated the flexibility of the Flex-Fuel Test Facility (gasifier/NGC Process section) to efficiently produce syngas from three different feedstocks, to condition the resulting raw syngas to meet the very stringent cleaning requirements of chemical synthesis applications (i.e., total S < 50 ppbv, total halides < 10 ppbv, particulate < 0.1 ppmw).

Consistent with the significance of the data developed in the PDU test program and the recommendations of conceptual process evaluations, GTI highly recommends the NGC Process development work enter into a process optimization phase. Specifically, four additional test campaigns

are proposed prior to undertaking further scale-up work and ultimately commercial-scale demonstration. The objectives of these tests are to:

- Optimize key process parameters:
  - sorbent feed rates
  - Sorbent sizes and size distributions
  - process operating temperatures
  - inlet contaminant levels
- Explore the envelope of these parameters both separately and in an integrated configuration
  - Develop the necessary data to extract quantitative design parameters for scaling up the bulk HCl removal performance (in the Stage I barrier filter-reactor simultaneously with ash), and the combined removal of sulfur and halide species to ppbv levels in the Stage II barrier filterreactor.
  - Extract quantitative information (filter cake permeability, cake thickness, portion of ash reaching the filter elements, etc) from the pressure drop data (based on ash flow rate and size distribution to the CF-R and filter cake properties, such as density, re-entrainment rate, etc.)
- Conduct integrated testing to demonstrate NGC Process suitability for "futuristic" IGCC and methanol synthesis applications.

In addition to process optimization, the proposed campaigns will provide another opportunity to perform additional mercury removal testing (using both the lower temperature and higher temperature sorbents and potentially other promising Hg sorbents), further advancing the syngas mercury capture technology base. Other facets of the process will also be explored including bulk HCl removal at higher temperature, continuous removal of ash/spent halide sorbent fines from the CF-R, etc.

## 2. PROGRAM OBJECTIVES

The Novel Gas Cleaning Program consisted of two phases: a Base Program phase and an Optional Program phase. The goal of the Base Program (completed in August 2001) was to provide the necessary data to verify the technical and economic feasibility of the Novel Gas Cleaning (NGC) Process. The scope of work in this first phase was dedicated most heavily to the laboratory testing of selected candidate sorbent materials for removal of reduced sulfur species (primarily H<sub>2</sub>S and COS) and halide species (primarily HCl) from simulated fuel gas mixtures. Experimental approaches were designed to closely simulate the gas/solid contact as well as the operating conditions in the NGC Process. Analytical techniques were developed for the measurement of sulfur and chloride species at trace levels to generate reaction conversion performance data. The most suitable desulfurization and dechlorination materials were identified and the optimum ranges of operating conditions determined for each stage of the NGC Process to achieve the target contaminant levels ( $H_2S < 60$  ppbv and HCl < 10 ppbv) in the cleaned fuel gas with acceptable conversion rates (i.e., sorbent feed rates). Laboratory test results were reported in the Base Program Final Report.<sup>1</sup>

The main goal of the second phase of the Novel Gas Cleaning Program (Optional Program) was to conduct a proof-of-principles demonstration of the NGC Process on a GTI carbonaceous fuel gasifier producing raw fuel gases representative of commercial gasifier practice, to confirm technical and economic feasibility of the process. An important objective of the Optional Program is to verify, at the PDU scale, the laboratory test results obtained in the Base Program with respect to operating temperatures for Stages I and II of the NGC Process, sorbent selections, sorbent/contaminant ratios, and ability of the process to accomplish very stringent contaminant levels in the syngas (i.e., particulates < 0.1 ppmw, total reduced sulfur compounds ( $H_2S$ , COS) < 60 ppbv, and total halides (mainly HCl) < 10 ppbv). In addition to particulates and sulfur and halide species, efforts have also been devoted in the Optional Program to evaluate the potential of incorporating mercury control into the Novel Gas Cleaning Process, with the objective of reducing mercury (mainly elemental mercury,  $Hg^0$ ) concentration in the syngas to less than 0.01 ppbv, corresponding to mercury removal efficiencies > 99%.

The Optional Program PDU test results, and the reaction rates and conversion factors extracted from the results, were used as the basis for an update of the commercial process performance and economics (Optional Program Final Report, Volume I). The fuel gas cleanup process technical and economic feasibility were estimated, and the areas of greatest performance and economic issues identified.

## **3. BACKGROUND**

Much of the hot gas cleanup research and development efforts have been conducted within the context of the Integrated Gasification Combined Cycle (IGCC), focusing predominantly on the development of regenerable metal oxide sorbents for the removal of reduced sulfur compounds (mainly hydrogen sulfide (H<sub>2</sub>S) and carbonyl sulfide (COS)) at high temperature and pressure. The objective in this application is to reduce the H<sub>2</sub>S concentration in the fuel gas to less than 20 ppmv to satisfy gas turbine requirements. In recent years there has been significant interest in extending the hot gas cleanup capabilities so that the cleaned fuel gas becomes suitable for a wide range of applications, including fuel cells and production of chemicals and transportation fuels, in addition to electric power generation via the IGCC technology. These applications require the removal to near-zero levels, of the sulfur-, nitrogen-, alkali-, and chlorine-containing gas emissions and fine particulate matter (PM<sub>2.5</sub>) caused by fuel bound constituents that are naturally present in many forms in a variety of carbonaceous materials which are useful as feedstocks to gasification processes. When the IGCC system is applied to creating synthesis gas for production of liquid products (e.g., methanol, F-T liquids), these species can be poisonous to

downstream processing units and may be contaminants in the high purity products. HCl in particular is a significant contaminant that causes severe deterioration to various fuel cell materials.<sup>2</sup>

Commercial technology is available to clean coal-gasification hydrocarbon gases to the stringent levels required for these demanding applications. The prevalent commercial gas cleaning process, capable of achieving the very stringent gas cleaning requirements for these types of applications, is based on the "Rectisol" gas desulfurization technology. The Rectisol process uses refrigerated methanol absorption of sulfur species and is expensive to build and operate, consuming extensive power. Commercial gas cleaning processes also apply wet, low-temperature removal of halides, particulates, and other contaminants, resulting in extensive water treatment requirements.

Three factors inherent in conventional, low-temperature fuel gas cleaning technologies result in lower power plant efficiencies and higher equipment costs:

- Nearly all of the water vapor in the fuel gas is condensed out, and is typically later replaced in a fuel gas humidification process, resulting in significant plant energy loss
- The low-temperature, sulfur absorption processes used remove a significant portion of the fuel gas CO<sub>2</sub> content along with the sulfur species, reducing the fuel gas mass flow and making sulfur recovery more energy intensive and expensive.

The process condensate streams generated require considerable processing to effectively remove contained contaminant salts and gases, increasing plant complexity and cost.

Several plant availability issues have been reported that relate to conventional, low temperature fuel gas cleaning:

- Volatile metal species in the cleaned fuel gas, in the form of iron and nickel carbonyls, are reported in some IGCC plants to result in deposition and corrosion in the gas turbine, and conventional fuel gas cleaning provides no protection from these metal carbonyls that are expected to form primarily at low temperatures within the fuel gas cleaning equipment.
- Particulate generated by fuel gas piping corrosion is reported in some IGCC power plants to reduce availability, resulting from gas turbine erosion and deposition, and has been dealt with in some IGCC power plants by adding a final fuel gas filter to protect the gas turbine from such particulate.
- The process condensate streams represent highly corrosive environments for process equipment and result in reduced power plant availability.

New, more economical technologies are needed for stringent gas cleaning duty if coal is to become competitive with cleaner fuel sources, such as natural gas, in U.S. Department of Energy advanced, near-zero emission, efficient multi-production energy plants.

To address this need, Siemens Power Generation, Inc. (Siemens) and the Gas Technology Institute (GTI) have been developing a novel process compatible with known gasifier systems that promises, in a pre-combustion treatment, to prepare carbonaceous feedstock-based fuel gases or syngases for use in power generation applications with gas turbines or with fuel cells, or for co-production of power with chemicals or transportation fuels. This process can be applied as an add-on cleaning step following a near-commercial, state-of-the-art hot gas polishing system (based on entrained-bed transport reactor technology, for example). The process can also be a stand-alone fuel gas cleanup system, depending on the carbonaceous fuel properties, the extent of in-gasifier cleaning, and the cleanup requirements of the intended application. This novel process specifically addresses sulfur, chloride, mercury, and particulate removal to meet the most stringent requirements (total sulfur (mainly H<sub>2</sub>S and COS) < 60 ppbv, halide (mainly HCl) < 10 ppbv, Hg < 0.01 ppbv, and particulate < 0.1 ppmw). These are contaminant levels below detection limits of conventional measurement instrumentation. In addition, various options have been conceptualized to incorporate the removal of other contaminants, such as ammonia (NH<sub>3</sub>), into the NGC Process. The NGC Process configurations, its barrier-filter reactor equipment, its performance goals, and its potential merits relative to commercial gas cleaning technologies were described in Volume I of this report.

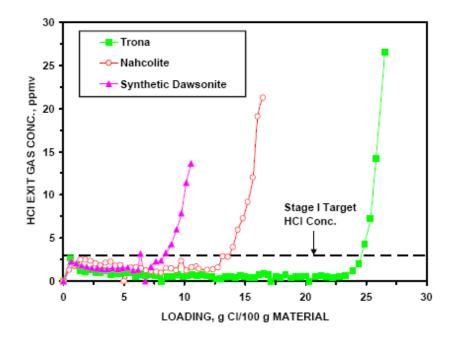
# 4. LABORATORY SUPPORTING STUDIES

The Novel Gas Cleaning Process concept was previously described in detail.<sup>3,4</sup> The rationale for the technical approach adopted, laboratory simulation of the process and operating conditions, sorbent/material requirements and selection, and reactor design and testing procedures were also outlined. Laboratory gas sampling and measurement procedures using gas and ion chromatography techniques were described and their reliability and accuracy were demonstrated.<sup>5,1</sup> Incorporation of mercury removal into the process and laboratory selection of sorbents and test evidence of sorbent performance capabilities have also been addressed in a dedicated publication.<sup>6</sup> A brief summary of the Base Program work scope and findings is provided below, as well as a concise summary of the mercury-related work (i.e., laboratory component of Task 6).

## 4.1 Sulfur and Halide Removal

In the first development phase, laboratory testing at GTI focused on the identification of suitable sulfur and halide sorbents and operating conditions for Stage I and Stage II of the NGC Process. This small-scale laboratory testing was also performed to provide evidence of the capability of the process to reach its stringent gas cleaning goals under operating conditions closely simulating process requirements, and also to explore the sensitivity of the sorbent performance to the major process parameters. This work systematically evaluated suitable Cu-, Fe-, Mn-, and Zn-containing fines and Na-containing fines as desulfurization and dechlorination materials, respectively, for both stages of the NGC Process. The materials selected for both stages of the process, their chemical and physical characteristics, their desulfurization or dechlorination performance in a packed-bed reactor, and the performance of physical mixtures of leading desulfurization and dechlorination materials were previously discussed in detail.<sup>5</sup> Consistency of experimental data was also demonstrated based on product gas analyses using gas and ion chromatographs (i.e., breakthrough curves), chemical analyses of spent samples from different locations in the sorbent bed, and X-ray diffraction (XRD) analyses. Based on thermodynamic simulations and analyses, guidelines were developed to rationalize the experimental work and confirm or explain the results obtained.

Tests with individual sorbents, and mixtures of sulfur and halide sorbents were conducted in simulated coal syngas containing individually H<sub>2</sub>S, HCl, or mixtures of these contaminants. These parametric tests were conducted at near-atmospheric pressure in a packed-bed reactor facility. The laboratory tests used a simulated coal-based syngas composition passing through thin packed beds of sorbents operated at face velocities representative of sorbent filter cakes in the NGC Process syngas polishing filter-reactors. Typical breakthrough data, such as shown in Figure 3, comparing HCl removal performance (efficiency and effective chloride capacity) of candidate Stage I dechlorination materials, were collected to measure the sorbent reaction performance. Sectional analysis of the packed bed sorbent reaction products to confirm the reaction mechanisms and conversions were included. The laboratory test procedures, equipment, and test results have been previously described.<sup>5</sup>



**Figure 3 - Performance Comparison of Stage I Dechlorination Minerals** 

The laboratory testing identified a specific set of zinc-based and sodium-based sorbents having the capability of meeting the process performance requirements for each stage and demonstrated this performance potential through laboratory test simulations. The sorbent characteristics and selected stage operating conditions are:

- Stage I temperature: 499°C (930°F),
- Stage I sulfur sorbent type: IGTSS-362C (Zn/Ti mole ratio 1.5),
- Stage I halide sorbent type: trona (Na<sub>2</sub>CO<sub>3</sub>• NaHCO<sub>3</sub>• 2H<sub>2</sub>O),
- Stage I sorbents size distribution: -325 mesh, mass-mean diameter about 20 μm,
- Stage II temperature: 288°C (550°F),
- Stage II sulfur sorbent type: G-72E (70 wt% Zn),
- Stage II HCl sorbent type: G-92C (6.4 wt% Na),
- Stage II sorbents size distribution: -325 mesh, mass-mean diameter about 20 μm.

The Stage I sulfur sorbent, IGTSS-362C, is a manufactured, zinc-based sorbent previously developed by GTI in granular form for high-temperature H<sub>2</sub>S removal. The Stage I halide sorbent, trona, is an inexpensive, commercially available, natural mineral. The Stage II sorbents G-72E (zinc-based) and G-92C (sodium-based) are catalyst pellet materials that are commercially available through Süd-Chemie.

Uncertainties exist in making gas contaminant measurements as low as 60 ppbv for  $H_2S$  and 10 ppbv for HCl, and a critical element of the program was the development of reliable procedures and equipment to make these measurements. State-of-the-art equipment, sampling and measurement and operating procedures were utilized by GTI to minimize and account for contaminant losses and background contaminant levels. Ion Chromatography was successfully used to make HCl measurements down to 10 ppbv. Stage I sulfur species contents down to 1 ppmv were reliably measured by a gas chromatograph equipped with a flame photometric detector (FPD). The Stage II sulfur species were

measured by a special gas chromatograph technique, but could only achieve a detection limit of 85 ppbv, compared to the target of 60 ppbv. This was considered acceptable for the Base Program laboratory screening and verification tests. GTI procured a trace sulfur system featuring the Varian 3800 Gas Chromatograph with built-in high performance sample concentrator and a sensitive pulsed flame photometric detector (PFPD). This system has a detection limit of 30 to 50 parts-per-trillion (pptv). It would be used for speciated and total sulfur analyses in the ultra-cleaned syngas during the testing campaigns planned in the Flex-Fuel Test Facility.

### 4.2 Mercury Removal

To incorporate mercury control into the NGC Process, GTI, in collaboration with Siemens, evaluated much of the prior and ongoing scientific and engineering studies on mercury removal in oxidizing as well as reducing gases, made many contacts with sorbent/catalyst manufacturers, and compiled a list of potential mercury sorbent materials for the syngas application. Candidate sorbents for meeting the stringent mercury removal requirements of commercial syngas applications include materials with potential to amalgamate with mercury (disposable as well as regenerable dispersed metal-based sorbents such as Ag, Au, Pt, Pd, Cu, etc.) and physical adsorbents, including chemically-promoted activated carbons, metal oxides, metal sulfides, halide salts, amended silicates, chlorinated sorbents, noble metals, fly ashes, zeolites, and calcium compounds.

On the basis of this assessment a primary list of sorbents (bulk and supported) was identified, comprising 15 materials based on the oxides of copper, manganese, molybdenum, and zinc, as well as mixed oxides, by-product materials, and activated carbons. The various sources for copper-based materials (bulk as well as supported sorbents) include a copper sulfide from a Canadian mine, Süd -Chemie, ALCOA, and previous GTI sulfur sorbent development programs. Mixed-oxide materials were obtained primarily from Johnson Matthey (MJ), and are marketed commercially as low-temperature shift conversion catalysts, or for sulfur and mercury removal at temperatures < 95°C (203°F). These consist of copper compounds (CuO and Cu<sub>2</sub>(CO<sub>3</sub>)(OH)<sub>2</sub>), zinc oxide (ZnO), and alumina (Al<sub>2</sub>O<sub>3</sub>). To promote the performance of these mixed-oxide materials for mercury removal from hydrocarbon streams and fuel gases at elevated temperatures, "especially sulfided" versions of these materials were also procured from JM. Molybdenum- and manganese-based materials (MoO<sub>3</sub>/TiO<sub>2</sub> and MnO/Al<sub>2</sub>O<sub>3</sub>) were obtained from the Millennium Inorganic Chemicals Co. in France and Chemetals in Baltimore, respectively. The "Norit Americas Darco FGD" activated carbon was selected to serve as a baseline sorbent in the laboratory portion of this work, and also as a potentially effective mercury sorbent at low temperature (38 to 121°C; 100 to 250°F) for chemicals synthesis applications. This material has been widely used in U.S. DOEsponsored demonstration programs for flue gas mercury removal via sorbent injection. Finally, an additional sulfur-impregnated activated carbon was obtained from Calgon Corporation in the form of 4mm extrudates containing 10-18% S, with reported effectiveness for mercury removal from natural gas, air, and by-product hydrogen streams.

GTI designed and constructed a totally enclosed reactor facility and procured a state-of-the-art gas-phase mercury analyzer (PS Analytical Sir Galahad II). Using the "Norit Americas Darco FGD" activated carbon material as a baseline sorbent, GTI validated the reactor system and associated mercury sampling and analysis procedures. All procured materials were evaluated systematically in stages, first using Hg<sup>0</sup>-N<sub>2</sub> mixtures, and then using fuel gas mixtures simulating the Texaco gasifier gas composition. Promising candidates have also been subjected to parametric testing in the 38-288°C (100-550°F) temperature range, under a wide range of other key operating conditions. Over 60 screening/scoping tests were completed. A typical breakthrough curve, obtained with a promising formulation, is shown in Figure 4. This material, designated as GTI-Hg-S9, was found to exhibit acceptable mercury removal performance in the 149-204°C (300-400°F) temperature range, achieving effective mercury capacity of approximately 0.015 wt.%. GTI worked with JM (formerly Synetix) to produce about 12.5 kg (27.5 lb) of this material in the -325 mesh size range for pilot-scale testing in the Flex-Fuel Test Facility, where it

would be injected into a coal-derived syngas upstream of the Stage II barrier filter-reactor in the NGC Process section. GTI also initiated efforts with JM to improve the performance of this material at the higher Stage II operating temperature.

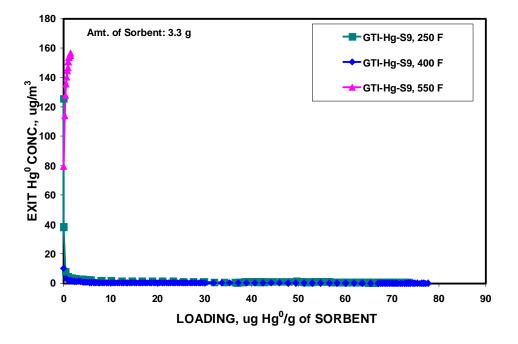


Figure 4 - Hg<sup>0</sup> Removal Performance of the GTI-Hg-S9 Mixed Oxide Sorbent at 121-288°C

To gain insight into the performance of these mercury sorbents, selected materials were subjected to chemical analysis and physical characterization, including XRD analysis. As indicated in Table 2, the GTI-Hg-S9 and GTI-Hg-S10 pre-sulfided mercury sorbents have high surface areas (45 and 38 m<sup>2</sup>/g), about one order of magnitude higher than those of typical regenerable sulfur sorbents (such as the IGTSS-179 copper-based sorbent listed in Table 2) manufactured using granulation or spray drying techniques. However, these surface areas are well within reach using a proprietary sorbent manufacturing technique developed at GTI (see IGTSS-362 zinc titanate sorbent properties in Table 2). Accordingly, GTI expects that it may be quite feasible in a future effort to formulate effective mercury sorbents using its modified sol-gel based preparation technique.

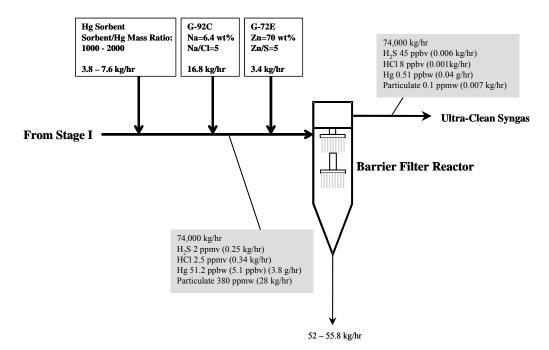
Material balance estimations were previously made for both stages of the NGC Process, in conjunction with a gasification plant processing 909 tonnes/day (1000 tons/day) of coal. The flows are roughly equivalent to those that would represent an advanced, 150 MWe fuel cell plant. Figure 5 shows a "conservative-case scenario" where G-92C and G-72E, dechlorination and desulfurization materials, respectively, are each assumed to achieve only 20% conversion (i.e., Zn/S=5 and Na/Cl=5).

Chemical & Physical	GTI-Hg-S8	GTI-Hg-S9	GTI-Hg-S10	GTI-Hg-S11	IGTSS-179	IGTSS- 362
Properties						
Aluminum, wt%	8.71	8.56	5.31	2.69	17.9	
Copper, wt%	34.5	32.4	26.4	38.5	35.8	
Zinc, wt%	20.4	19.9	13.8	7.11		30.2
Manganese, wt%					11.3	
Titanium, wt%					0.54	33.1
Carbon, wt%			5.95	4.63		
Hydrogen, wt%			2.0	1.14		
Sulfur, wt%	< 0.01	14.3	16.7	< 0.01		
Bulk density, g/cm <sup>3</sup>	2.11	2.33	1.70	2.16	1.93	1.43
Particle (Hg) density (pb), g/cm <sup>3</sup> *	2.11	2.33	1.67	2.16	3.65	2.56
Skeletal density ( $\rho_a$ ), g/ cm <sup>3</sup>	4.16	3.48	2.93	3.47	4.17	4.17
BET Surface Area, m <sup>2</sup> /g	88.2	71.1	35.2	30.4		47.2
Porosity, %**	49.16	32.85	42.94	37.70	12.5	38.7
Hg Surface Area, m <sup>2</sup> /g	76.97	44.79	38.33	24.99	5.21	41.2

Table 2 - Physical and Chemical Characteristics of Selected Mercury and Sulfur Sorbents

## \*Corrected for inter-particle void

\*\*Calculated based on corrected values as (1 - pb/pa)\*100, or equivalently as pb\*(Hg Pore Volume)\*100



### Figure 5 - Material Balance for Stage II Barrier Filter-Reactor in the Ultra-Clean Process with Additional Mercury Control (Chemical Synthesis Application, 550°F)

Mercury input to the Stage II Barrier Filter-Reactor of the NGC Process was based on a coal mercury content of approximately 100 ppbw (0.1 ppmw), the average level for different types of U.S. coals. A concentration of 100 ppbw in the coal is equivalent to 51.2 ppbw (5.1 ppbv) in the syngas. The mass balance around the Stage II Barrier Filter-Reactor is shown in Figure 5. Based on the estimate of 1000-2000 sorbent-to-Hg<sup>0</sup> mass ratio, approximately 3.8 to 7.6 kg of sorbent is needed to remove 99% of the mercury, as indicated. The mercury loading based on the mass balance can be calculated to be approximately 500-1000  $\mu$ g Hg<sup>0</sup>/g of sorbent (i.e., 0.05 to 0.1 wt% Hg retention capacity). This is quite

reasonable considering that Eastman Chemical Co. has already demonstrated economic mercury removal from syngas at low temperature using activated carbon with effective capacity of about 0.4 wt.%.<sup>7,8</sup> Moreover, carbon-to-mercury weight ratios of 3,000:1 to 100,000:1 have been reported for mercury control in coal-fired utility flue gases via powdered activated carbon injection.<sup>9</sup>

For the promising GTI-Hg-S9 sorbent, according to the sorbent manufacturer (JM), the cost would be about  $10,000-15,000/m^3$ . This cost includes sorbent procurement from a JM facility in the U.S. and pretreatment (i.e., sulfidation) in a facility in the United Kingdom. The delivered cost for the sorbent would be about 10 to 15/kg (\$4.5 to \$6.8/lb), close to that of zinc titanate.

## 5. NGC PROCESS SECTION CONCEPTUAL DESIGN

Preparation of the NGC Process section was conducted in parallel with the Flex-Fuel Test Facility design and construction activities. This included designing a suitable process configuration and specifying, procuring, and installing the necessary pieces of equipment. The final NGC Process section layout, shown schematically in Figure 6, was determined after evaluation of several options and redirection of the program goals to focus on the more challenging aspects of the project, i.e., to achieve very stringent contaminant removal targets at the ppbv level. This part of the plant consists of two major sections: Conditioning and Testing. In the Conditioning section, the raw gasifier gas is treated to meet the requirements of the NGC Process Testing section (i.e., Stage II). The Conditioning section, however, does incorporate one of Siemens' barrier filters primarily to control syngas particulates, but also to remove the bulk of halides (i.e., HCl). Therefore, this first barrier-filter reactor represents Stage I in the NGC Process base configuration (i.e., Figure 1).

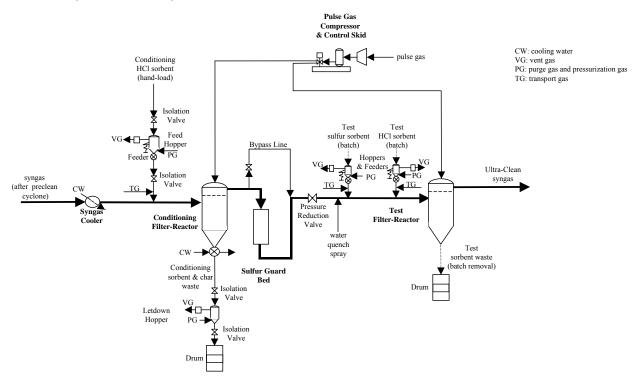


Figure 6 - Ultra-Clean Bench-Scale Test Facility Process Flow Diagram

An initial evaluation of the Novel Gas Cleaning Process pilot-scale test system was performed, providing for estimates of equipment duties and auxiliaries needs. A set of assumptions was used to allow

the development of process material and energy balances and preliminary sizing of the equipment components. These assumptions were conservative to allow appropriate equipment design capacities to be selected.

## 5.1 Design Assumptions

## 5.1.1 Syngas Delivery Conditions

- Gasifier coal: Montana Coal (0.62 moisture-free wt% sulfur; 8.3 moisture-free ash)
- Gasifier: GTI air-blown fluid bed
- Gasifier desulfurization: none
- Inlet syngas composition at exit of secondary cyclone:

$H_2$	13.83 mole%
CH <sub>4</sub>	2.21
CO	15.73
$CO_2$	10.5
H <sub>2</sub> O	12.21
$N_2$	45.37
Sulfur (H <sub>2</sub> S plus COS)	941 ppmv
Halides (HCl plus HBr)	100 - 500
Fuel nitrogen (NH <sub>3</sub> plus	HCN) 1240 ppmv
Hg 2 ppbv (estimate)	

- Syngas particulate content at secondary cyclone exit: 21,250 ppmw char (nominal GTI estimate) 31,250 ppmw (maximum expected)
- Syngas temperature at secondary cyclone exit: 1634°F
- Syngas pressure at secondary cyclone exit: 285 psia
- Syngas flow rate at secondary cyclone exit: 3300 lb/hr for all tests

The  $H_2S$  and COS contents of the syngas are uncertain and are initial estimates, but the sulfur content of the syngas does not influence any of the test equipment designs other than the guard bed desulfurizer unit. The halide and particulate contents currently have very broad ranges of minimum and maximum expected values. Their estimated syngas contents may increase or decrease as the process estimates are improved. Other components listed above (NH<sub>3</sub>, HCN) are included for future interest.

## 5.1.2 Test Conditions

- Syngas cooler outlet temperature: 830°F
- Halide removal in Conditioning Filter-Reactor using trona sorbent (maximum Stage II inlet halide of 5 ppmv; minimum of 1 ppmv)
- Guard Bed sulfur removal achieves > 99%, with bypass valve fully shut
- Maximum Stage II inlet  $H_2S + COS = 5$  ppmv; minimum 1 ppmv
- Syngas pressure at Test Filter-Reactor inlet: 215 psia
- Test Filter-Reactor inlet temperature of 550°F (after sorbent injection)
- Target Stage II Test Filter-Reactor outlet  $H_2S + COS = 60$  ppbv
- Target Stage II Test Filter-Reactor outlet halide = 10 ppbv

- Maximum test run duration in syngas:
  - Conditioning Filter-Reactor, 120 hr
  - Sulfur Guard Bed, 120 hr
  - Test Filter-Reactor, 96 hr
  - Number of test campaigns, 3

## 5.1.3 Sorbents

Conditioning halide sorbent: Trona (Na2CO3•NaHCO3•2H2O)

- 50 mol% H<sub>2</sub>O
- 25 mol% sodium carbonate
- 25 mol% sodium bicarbonate
- maximum molar feed rate 10 times stoichiometric; minimum 2.0

## Sulfur Guard Bed sorbent: BASF Catalyst R 5-12

- Approx. 95% highly activated ZnO extrudates
- sorbent utilization 50%

## Stage II sulfur sorbent: G-72E

- 90.92 mol% ZnO
- 2.48 mol% CaO; 6.60 mol% Al<sub>2</sub>O<sub>3</sub>
- maximum molar feed rate 10 times stoichiometric; minimum 2.0

## Stage II Halide sorbent: G-92C

- 15.96 mol% sodium oxide
- 84.04 mol% Al<sub>2</sub>O<sub>3</sub>
- maximum molar feed rate 10 times stoichiometric; minimum 2.0

## 5.1.4 Pressure Drops and Heat Losses

Estimates were included for component and piping pressure drops and heat losses. The following assumptions were made.

Refractory-lined pipe lengths for pressure drop estimates (3" ID, refractory-lined pipe):

- Secondary cyclone to syngas cooler inlet: 3 ft
- Syngas cooler to HCl conditioning sorbent injection point: 2 ft
- HCl conditioning sorbent injection point to Conditioning Filter-Reactor inlet: 5 ft
- Conditioning Filter-Reactor exit to Sulfur Guard Bed inlet: 5 ft with no insulation to promote syngas cooling
- Sulfur Guard Bed exit to Stage II Trim Cooler point: 5 ft
- Trim Cooler to sulfur sorbent and HCl sorbent injection points: 3 ft
- Sorbent injection points to Test Filter-Reactor: 5 ft

Component heat loss estimates:

- Ambient temperature 70°F
- Equivalent, overall insulated pipe estimated as a function of refractory thickness:
  - 1634°F: refractory thickness 4 in. and overall heat transfer coefficient of 2 Btu/hr ft<sup>2</sup>°F
  - 550-950°F: refractory thickness 3 in. and overall heat transfer coefficient 1.5 Btu/hr ft<sup>2</sup> °F
  - Non-insulated pipe length overall heat transfer coefficient of 20 Btu/hr ft<sup>2</sup> °F
  - Filter-Reactor heat losses estimated as pipe 10 ft long with overall heat transfer coefficient of 4.5 at Stage II conditions

Component pressure drops:

- Syngas cooler heat exchanger gas-side pressure drop estimated to be 0.5 psi
- Filter-Reactors average pressure drop about 3% of inlet pressure (7 psi used)

- Sulfur Guard Bed pressure drop estimated to be 5 psi

## 5.1.5 Auxiliaries

- Pressurization gas rate: 1.1 times feed sorbent voidage flow, based on bulk voidage of 80%
- Solids transport gas rate: 3/8" tubing (20 gage) with gas velocity of 50 ft/s, results in 105 lb/hr transport gas rate per feed line
- Waste solids purge gas rate: 1.1 times waste solids voidage flow, based on bulk voidage of 80%; value fixed at maximum level
- Filter pulse gas rate: 0.1 lb of gas per pulse per candle; 14 candles in vessel; Conditioning Filter-Reactor pulse cleaned 6 times per hour, and Stage II Filter-Rector pulse cleaned 1 time per hour
- Pulse gas supplied to test facility at 400 to 600 psia
- Pulse gas source nominal pressure 600 psi above operating pressure (900 psia)
- Pulse gas compressor adiabatic efficiency 75%
- Test facility nitrogen composition is 98 vol% N<sub>2</sub>, 2 vol% O<sub>2</sub>
- Cool waste solids from Conditioning Filter-Reactor to 250°F (if force cooling is needed)
- Cooling water and quench water available at  $70^{\circ}$ F; delta-temperature =  $10^{\circ}$ F

## 5.2 Computed Results

## 5.2.1 Filter-Reactor Conditions

Conditioning	Filter-Reactor
Conditioning	1 mor-reactor

tioning i neer reductor	
Inlet temperature (°F):	830
Outlet pressure (psia):	277
Outlet temperature (°F):	770-808
Inlet gas flow (acfm):	107
Face velocity (ft/min):	2.7
Inlet dust load (ppmw):	27,500-42,000 (including trona)
Halide removal (%):	98.5 (min) - 98.9 (max)

## Test Filter-Reactor

Inlet temperature (°F):	550 (after sorbent injection)
Outlet temperature (°F):	533
Inlet gas flow (acfm):	123
Face velocity (ft/min):	3.1
Inlet dust load (ppmw):	38 (min) - 690 (max)
Sulfur removal (%):	94.2 (min) - 97.7 (max)
Halide removal (%):	99.0 (min) - 99.8 (max)

## 5.2.2 Feed System Requirements

Conditioning halide sorbent feed system (trona -36 lb/ft	<sup>3</sup> bulk density <u>)</u>
minimum feed rate (lb/hr; ft <sup>3</sup> /hr):	2.0; 0.056
maximum feed rate (lb/hr; ft <sup>3</sup> /hr):	34.4; 0.96
Total feed per test campaign (lb; ft <sup>3</sup> ):	40-4,128; 7-115
Total feed for program (3 test campaigns) (lb):	720 -12,384

Test halide sorbent feed system (G-92C - 36 lb/ft <sup>3</sup> bulk density)			
minimum feed rate (lb/hr; ft <sup>3</sup> /hr):	0.092; 0.0025		
maximum feed rate (lb/hr; ft <sup>3</sup> /hr):	2.3; 0.064		
Total feed per test campaign (lb; ft <sup>3</sup> ):	8.8-221; 0.24-6.1		
Total feed for program (3 test campaigns) (lb):	26 - 663		

Test sulfur sorbent feed system (G-72E – 61 lb/ft³ bulk density)minimum feed rate (lb/hr; ft³/hr):0.04; 0.0007maximum feed rate (lb/hr; ft³/hr):0.28; 0.0046Total feed per test campaign (lb; ft³):3.8-26.9; 0.06-0.44Total feed for program (3 test campaigns) (lb):12 - 81

## 5.2.3 Sulfur Guard Bed

	At minimum	At maximum
	flow conditions	flow conditions
Bypass (% of syngas flow)	0	0
Guard bed sulfur removal (%)	99.6	99
Outlet sulfur content (ppmv)	1	2.7
ZnO consumption (lb/hr)	6.2	6.2
Total ZnO consumed in run (lb)	744	744
Inlet temperature (°F)	734	720

Total BASF guard bed ZnO sorbent consumed in test program about 2150 lb.

## 5.2.4 Waste Handling System Requirements

Sorbent wastes bulk density assumed 30 lb/ft3; gasifier char bulk density assumed 20 lb/ft<sup>3</sup>

Condit	ioning waste system		
	minimum rate (lb/hr; ft <sup>3</sup> /hr):		69; 3.5
	maximum rate (lb/hr; ft <sup>3</sup> /hr):	125	5; 6.3
	Total per test campaign (lb; ft <sup>3</sup> ):		8,280-15,000; 420-750
	Total for program (3 test campaigns) (lb	<b>)</b> :	25,000 - 45,000
Test wa	aste system		
	minimum rate (lb/hr; ft <sup>3</sup> /hr):		0.14; 0.005
	maximum rate (lb/hr; ft <sup>3</sup> /hr):	2.5	; 0.083
	Total per test campaign (lb; ft <sup>3</sup> ):		13-243; 0.43-8.1
	Total for program (3 test campaigns) (lb		
5.2.5	Axiliaries		
Nitroge	en		
-	transport maximum (lb/hr):	315	5
	pulse cleaning gas maximum (lb/hr):15		
	purge gas maximum (lb/hr):	4	
	pressurization gas (lb/hr):	1	
	Total Nitrogen (lb/hr):	335	5
Coolin	g Water		
	syngas cooling (gpm):	185	5
	waste solids cooling max (gpm):	1.8	
Process	s Water		
110005	syngas quench max (gpm):	0.2	5
		0.2	5
Electric			
	Pulse gas compressor max (kW):	0.2	
	Screw conveyor max (kW):	0.8	5 (if used)

### 5.3 Conclusions

#### Conditioning HCl Sorbent (Trona) Feed System

This pressurization and feed system will use a batch, pressurization and feed hopper, with isolation valves, having a maximum storage capacity of about 144 lb, with a total bulk solids volume of 4  $ft^3$ . Its design pressure is 300 psia. It will feed HCl sorbent (trona) to the Conditioning Filter-Reactor during testing. The feeder must control and measure the feed rate with 10% accuracy over a range of 2.0 to 35 lb/hr using a volumetric feeder to simplify construction. Basis for sizing is 36 lb/ft<sup>3</sup> measured bulk density. The refill time for this feed system ranges from a minimum of once every 10 hours to once per test campaign.

#### Conditioning Filter-Reactor

Drainage of the conditioning waste solids lock hopper must be accomplished periodically during conditioning filter vessel drainage shut-off. The maximum operating time that the Conditioning Filter-Reactor filter vessel can operate without vessel drainage is 5-9 hours. The new filter vessel, with design pressure of 300 psia, is used for this duty. The new filter vessel is nearly identical with the existing filter vessel, except that its hopper section storage capacity is enlarged (by about 10 ft<sup>3</sup>) to store solids during the testing should drainage be interrupted.

### Conditioning Waste Solids Handling System

A batch, 12-hour hopper, with isolation valves, will be used having a maximum capacity of 1500 lb, and a total volume of about 83 ft<sup>3</sup>. Its design pressure is 300 psia. This hopper should be placed directly under the Conditioning Filter-Reactor so that the solids storage capacity is available without intermediate transport equipment. An existing water-cooled screw conveyor may be used for transport and solids cooling from this hopper, if it is found that it can handle the cooling duty (solids cooling from 800 to 150°F, solids rate 828-1500 lb/hr (41-83 ft<sup>3</sup>/hr), cooling load 140,000-254,000 Btu/hr, cooling water 13.9-25.2 gpm). GTI assumes one-hour drainage time. GTI recommended reducing the holding time for this hopper to 6 hours to reduce its size.

#### Sulfur Guard Bed

A bypass valve having an operating range of 0 to 17% of the full syngas flow during testing is needed to control the test system inlet syngas sulfur content. This bypass valve expected to be fully shut during the Stage II testing, and all of the syngas will pass through the guard bed. The guard bed should be sized to achieve at least 99 sulfur removal ( $H_2S$  and COS) at maximum sulfur flow rate.

#### Pressure Reduction Valve

This valve, located after the Sulfur Guard Bed, will reduce the syngas pressure from 285 psia (maximum) at 850°F, to 215 psia at a maximum syngas flow rate of 3,323 lb/hr (105 acfm at inlet conditions).

#### Water Quench Spray

Process water will spray quench the syngas stream, at a maximum rate of 2 lb/hr, to cool the syngas to 550°F, measured at a location following sorbent injection, but before the Test Filter-Reactor inlet nozzle. Indirect cooling and drying of the syngas may be incorporated in the future.

### Test Sulfur Sorbent Feed System for Stage II

This existing pressurization and feed system is a batch, pressurization and feed hopper having a maximum capacity of about 60 lb and a total volume of about 1 ft<sup>3</sup>, based on the Stage II maximum feed rate. It will contain many hours of sorbent for the Stage II testing and refill will not be needed (217 - 1428 hours feed capacity). Its design pressure is 300 psia. The feeder must be able to feed with 10% accuracy

over the range of 0.04 to 0.3 lb/hr using a differential-weight feeder. Basis for volume is bulk density of 61 lb/ft<sup>3</sup>. The pressure vessel dimensions are 39-inch OD with 63-inch height.

#### Test HCl Sorbent Feed System for Stage II

This pressurization and feed system will be a batch, pressurization and feed hopper having a capacity of 144 lb and a total volume of 4.0 ft<sup>3</sup>. Its design pressure is 300 psia. The feeder mechanism must be able to feed with a 10% accuracy over the range of 0.09 - 2.3 lb/hr using a differential-weight feeder. Basis for volume is bulk density of 36 lb/ft<sup>3</sup>. The sorbent capacity of this feeder vessel will be 156 - 4,000 hours, so refill will not be required. The vessel will have identical design to that of the trona feed vessel.

#### Test Filter-Reactor

The existing filter vessel will be used for this duty. The existing filter vessel has a sorbent storage capacity of 81-1,500 hours during Stage II testing.

#### Test Sorbent Waste Handling System

The Test Filter-Reactor and drain pipe will hold the entire Stage II test campaign waste accumulation without drainage. A separate depressurization vessel is not needed. Heat losses through the vessel wall following system turndown should be sufficient and cooling water is not needed. Care must be taken to avoid condensation within the waste solids before drainage occurs.

#### Pulse Gas Compressor and Control System

The existing system will be adapted to serve the pulse cleaning needs of both filter vessels, with the Conditioning Filter-Reactor being pulse cleaned several times per hour, and the Test Filter-Reactor being pulse cleaned infrequently (< 1 per hour).

## 6. PREPARATION OF PILOT-SCALE FACILITIES

The main goal of the second phase of the Novel Gas Cleaning Program (Optional Program) has been to conduct a proof-of-principles demonstration of the integrated NGC Process on a  $\sim$  10-ton/day GTI coal gasifier to confirm technical and economic feasibility of the process. The original plan was to refurbish an existing GTI gasifier to generate syngas via coal gasification for the NGC Process testing. Project schedule, however, coincided with GTI's decision to construct a new carbonaceous fuel gasification facility (Flex-Fuel Test Facility) with capability to process about 10 tons of coal per day. Design and construction of the entire facility proceeded simultaneously.

#### 6.1 Flex-Fuel Test Facility

GTI recently constructed a unique test platform to address the need for more thorough evaluation of gasification technology as well as other equipment and processes that can be used to convert various feedstocks into synthesis gas.

Built with the support of the natural gas industry and the State of Illinois, the state-of-the-art Henry R. Linden Flex-Fuel Test Facility (Figure 7 and Figure 8) evaluates innovative gasification processes and facilitates the commercialization of advanced gasification and downstream end-use technologies. The FFTF is designed to evaluate a variety of gasifier technologies in a versatile platform for testing all ranks of coal and other solid fuels, including biomass. The facility's flexible design allows testing of a variety of gasification, gas cleanup, and gas processing schemes independently or as integrated systems.

The term Flex-Fuel has been used in the designation of the facility to indicate its ability to process a variety of feedstocks, primarily solids such as coal and biomass, and for its adaptability as an

R&D unit. The gasifier unit was designed such that it can be modified to accommodate additional process configurations or equipment, such as solids filtration systems or high-temperature pollutant removal vessels. For example, the lower gasifier section can be removed and replaced to simulate a RENUGAS® type gasifier. Similarly, the upper section of the gasification reactor can be modified to incorporate a hydrogen-selective membrane for enhanced and direct hydrogen production from coal, an effort currently in progress at GTI.<sup>10</sup>

Innovative sampling and analytical systems were designed and implemented in the facility to enable detailed systems evaluations. Simultaneous on-line analyses of gas compositions from raw gas concentrations to ultra-clean levels allow for near real-time assessment of the performance of integrated gasification and gas conditioning systems. Comprehensive and accurate diagnostic capabilities make costeffective testing of technologies and systems at the facility possible.

The heart of the Flex-Fuel facility is a U-GAS® type gasification reactor which uses heat and pressure to convert solid feedstocks such as coal and biomass into low to medium Btu-content gas. Following further processing, the produced fuel gas can then be used as fuel, purified to hydrogen, or used as feedstock for chemicals production. The Flex-Fuel gasifier is designed to operate at pressures up to 420 psig (30 bar) and temperatures up to 2000°F (1093°C), and can be used in conjunction with:

- Coal: 10 tons per day (tpd) with air, 20 tpd with oxygen
- Biomass: 20 tpd with air; 40 tpd with oxygen
- Downstream syngas clean-up and separation systems
- Advanced power conversion systems (fuel cells, small turbines, reciprocating engines)
- Coal to Synthetic Natural Gas (SNG)
- Syngas-to-Liquids conversion
- Hydrogen production
- CO<sub>2</sub> capture technology

The FFTF is intended for conducting research and validation of enabling technologies that have shown promise after laboratory and bench testing and need demonstration at a larger scale. These systems can be tested on either slipstreams from the gasifier or on the full gas stream on a dedicated basis or in conjunction with other users to reduce test costs. Novel gasification concepts can be tested at a variety of sizes in test campaigns typically conducted over a week period. Continuous long-duration testing is also possible.



Figure 7 – GTI's Newly-Commissioned Flex-Fuel Test Facility

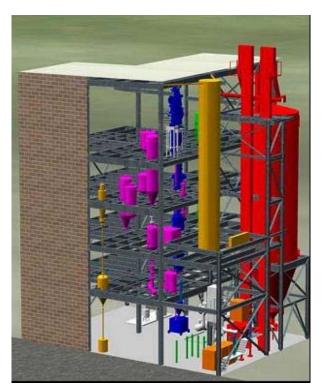


Figure 8 - Cutaway View of the FFTF Showing Solids Storage and Handling (Red), Gasification and Syngas (Blue), and NGC Process (Purple) Equipment

The FFTF includes an 11-inch (28 cm) diameter, adiabatic, single-stage fluidized-bed reactor system capable of gasifying 10 tons per day of coal in the air-blown mode at pressures up to 420 psig. This pilot plant facility includes equipment, process piping and controls for feed storage, transport and weighing, feed lock hopper pressurization and metering, air compression, fluidized bed gasification, ash and fines collection, depressurization, and weighing, and syngas cooling, cleaning, and flaring. Additional equipment, piping and controls provide utilities such as high pressure superheated steam, high-pressure nitrogen, demineralized high pressure quench water, process water, instrument air, and fire water.

The entire structure stands about 75 feet ( $\sim 23$  m) tall, with 5 decks of 1250 ft<sup>2</sup> (116 m<sup>2</sup>) of process area per floor. The process area houses the various pressure vessels, reactors, piping and instrumentation necessary to run the facility. The facility is wholly contained (Figure 7) except for electricity, natural gas, and water. On the north side of the structure is the fuel storage silo, where feedstocks are held until transferred into the process unit. The facility was designed to process 10 tons (9 tonnes) of coal or 24 tons (21.8 tonnes) of biomass per day. All major equipment is shown in Figure 9. This Process Flow Diagram (PFD) shows the major flow streams for the storage and unloading of the coal; coal, air and steam feeding to the gasifier; and syngas to the flare. The NGC Process section, fully integrated into the Flex-Fuel Test Facility, is also shown.

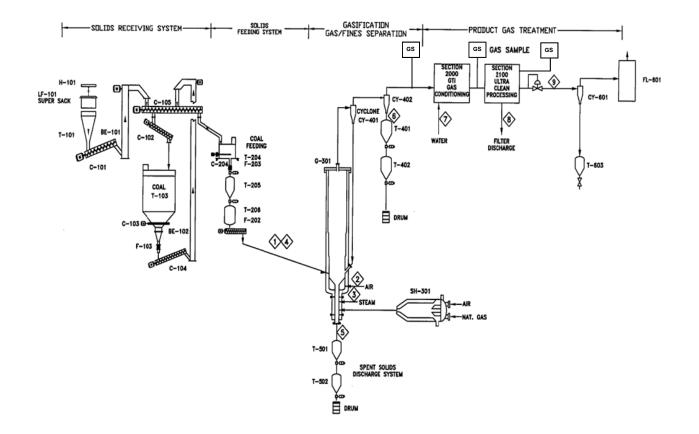


Figure 9 - Flex-Fuel Test Facility Process Flow Diagram

The FFTF solids receiving section receives feed materials from bulk storage bags, stores the feed in a storage silo, and transfers feed materials to the solids feeding section. Solid feed materials are dried and sized off-site and delivered to the plant by trucks in bulk storage bags and unloaded at the receiving area. The receiving area is enclosed with roof and retaining walls for weather. It is equipped with filtering ventilation for control of dust emission.

A forklift truck transfers coal to the loading hopper. The loading hopper is equipped with an open top and a conical bottom for top-fill and bottom-discharge operation. A screw feeder at the bottom of the loading hopper discharges the solids into a bucket elevator that conveys the solids into the storage silo.

The storage silo is an atmospheric cylindrical storage vessel designed for top fill and bottom discharge of fuel. The silo is sized to store a week's inventory of test fuel. It has a conical bottom with an apex angle of  $60^{\circ}$  and is equipped with a bin activator assembly to allow use with biomass fuels. The silo is continuously purged with nitrogen to prevent oxidation of the fuel in the bin during storage.

Coal is discharged from the silo with a screw conveyer and bucket elevator. A second screw feeder transfers the coal from the bucket elevator to the weigh hopper. Coal can be delivered to the weigh hopper from either the storage silo or directly from the loading hopper. This allows coal switching without the need to empty the silo.

The weigh bin is equipped with load cells to continuously monitor and record coal feed batch weights as they enter the lockhopper system. At a set interval, the coal from the weigh bin is discharged into the lock hopper via rotary feeder. The lock hopper is equipped with quick open/close valves for cyclic pressurization and depressurization.

When the lock hopper is loaded and pressurized, the bottom lock hopper valve opens and coal is emptied into the coal feed hopper. When the lock hopper is emptied, it is depressurized to ambient condition; the cycle of coal loading to the lock hopper is repeated. The frequency of this cyclical loading/unloading operation of lock hopper is typically between 1 - 2 cycles per hour.

The coal from the feed hopper is continuously discharged to a pressurized variable speed metering feed screw to control feed rate and is then pneumatically conveyed with nitrogen into the gasifier.

When configured for fluidized-bed gasification the coal feed is processed (sized and dried) to the following specifications:

- $100\% < \frac{1}{4}$  inch
- No more than 10% < 100 mesh
- No more than 5% surface moisture

For the NGC Process PDU testing, the FFTF was configured with a refractory-lined fluidized-bed gasifier based on GTI's U-GAS® coal gasifier design. In the fluid bed, coal reacts with steam and air to produce hydrogen (H<sub>2</sub>), methane (CH<sub>4</sub>), carbon monoxide (CO) and carbon dioxide (CO<sub>2</sub>), which together with nitrogen (N<sub>2</sub>) from the gasification air constitute the major elements of the gaseous fuel (syngas) product.

Syngas exits the top of gasifier, where the larger entrained particulates are captured in a cyclone and recycled to the gasifier through a dip leg to improve carbon conversion. The residual ash discharges from the bottom of the gasifier bed through the ash lock hopper system. The ash discharge rate is controlled by a terminal velocity mechanism having no moving parts. Adjusting the upward gas velocity through the ash discharge pipe controls the ash discharge rate. Ash is discharged to a surge hopper and then an ash lockhopper where the ash is depressurized. The ash is periodically discharged into a drum and the drum is weighed in order to monitor the ash discharge rate. A start-up heater is used to preheat the refractory in the gasifier during start-up and then startup fuel is fed to the gasifier (typically metallurgical coke) to complete the heat-up process and ignition of the fluidized bed.

The FFTF is equipped with two refractory-lined cyclones in series downstream of the gasifier. The first (primary) cyclone separates the solids from the product gas exiting the gasifier freeboard. The separated fines in the cyclone dipleg are re-injected into the gasifier. The second (secondary) cyclone separates the residual fines from the gas exiting the primary cyclone. The fines removed in the secondary cyclone are discharged into a surge hopper and lockhopper, and subsequently into a collection drum, which is weighed to monitor the fines discharge rate.

The product gas exiting the cyclone is cooled to 650 to 850°F (343 to 454°C) by a direct-spray water quench prior to being routed to either the NGC Process system or directly to the flare. This is a "dry" quench with all water vaporized into the syngas stream. The syngas is then depressurized through a backpressure control valve station to near atmospheric pressure. A final cyclone captures any particulate left in the gas before the gas is combusted in a flare.

As indicated earlier, the overall Flex-Fuel Test Facility configuration (Figure 9) includes the gasification section, the NGC Process section, and the exhaust gas handling section. The NGC Process section is further divided into two major sections: Conditioning and Testing. In the Conditioning section, the raw syngas is first conditioned by bulk particulate, halide, and sulfur removal to produce a syngas having contaminant contents and temperature representative of inlet conditions to Stage II of the NGC Process. The Testing section is dedicated to assessing the most challenging aspects of the NGC Process performance, i.e., removal of reduced sulfur and halide species to ppbv levels. Detailed description of the NGC Process pilot-scale configuration is provided in the following report sections.

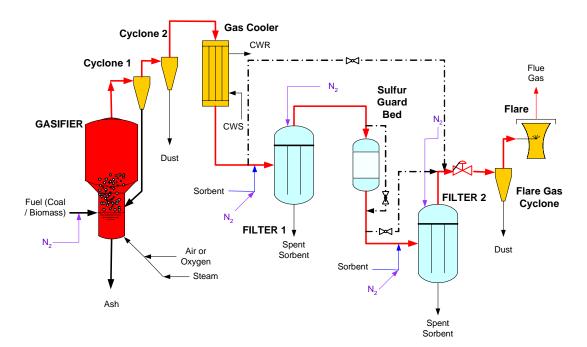


Figure 10 - Flex-Fuel Gasifier Facility Showing Ultra-Clean Process Section

## 6.2 Novel Gas Cleaning (NGC) Process Section

Preparation of the NGC Process pilot-scale test facility was carried out in parallel with the Flex-Fuel construction activities. This included designing, specifying, procuring, and installing various pieces of equipment for the NGC Process section, in concert with the requirements of the new gasification facility. The final NGC Process section layout was determined after evaluation of several options and redirection of the program goals to focus on the more challenging aspects of the project, i.e., to achieve very stringent contaminant removal targets at the ppbv level. This part of the plant consists of two major sections: Conditioning and Testing as shown in Figure 11, a piping and instrumentation diagram (P&ID) for the NGC Process section (a complete set of P&IDs is included in Appendix A of this report). In the Conditioning section, the raw gasifier gas is treated to meet the requirements of the NGC Process Testing section (i.e., Stage II). The Conditioning section, however, does incorporate one of Siemens barrier filters primarily to control syngas particulates, but also to remove the bulk of halides (i.e., HCl). Therefore, this first barrier-filter reactor represents Stage I in the NGC Process base configuration.

As shown in Figure 11, the NGC Process section consists of several major components:

### **Conditioning Section**

- Direct Spray Water Quench (DSQ)
- Conditioning Filter-Reactor (CF-R)
- Conditioning halide sorbent feed system (Stage I halide sorbent)
- Conditioning char and spent sorbent handling system
- Pressure letdown valve
- Sulfur Guard Bed and associated bypass line and pre-heater
- Syngas Trim Cooler

### Test Section

- Test Filter-Reactor, TF-R (for Stage II testing)
- Test sulfur, halide, and mercury sorbent feed systems (for Stage II testing)
- Test spent sorbent handling system

During a typical test, the gasifier is operated with coal in an air-blown mode to produce a syngas representative of commercial gasifier practice. Syngas off the Secondary Cyclone, at a temperature of about 1650°F, is cooled to about 830°F in the Direct Spray Quench tower. This stream is still relatively dirty, with a solids loading of about 2-3 wt%. Following flowrate measurement by an orifice flow meter and withdrawal of a gas sample for analysis, the syngas can be sent either towards the Conditioning Filter-Reactor inlet or bypassed towards the Tertiary Cyclone and then the Flex-Fuel Flare.

The cooled syngas from the Direct Spray Quench system is directed to the lower section of the Conditioning Filter-Reactor vessel (Figure 12), which contains 14 filter elements or candles (Figure 13). Filter elements (60-cm I.D., 1.5-m long Schumacher Dia Schumalith, clay-bonded silicon carbide) are mounted seven (7) on each of two semi-cylindrical plenums. The bulk dechlorination sorbent, trona (or nahcolite as an alternative sorbent), is metered from a 4-ft<sup>3</sup> hopper into the syngas immediately upstream of the Conditioning Filter-Reactor, by a loss-in-weight feeding system (Figure 14, Figure 15) enclosed in a pressure vessel. Trona, in the form of very fine powder (~ 20  $\mu$ m average particle size), is added at rates ranging from 2 to 35 lb/hr. The bulk halide sorbent is moved into the syngas stream by dilute-phase transport in nitrogen, which picks up the sorbent under the loss-in-weight feeding vessel de-pressured, the sorbent supply in the hopper replenished, the sorbent feeding vessel re-sealed and re-pressured, and sorbent addition resumed.

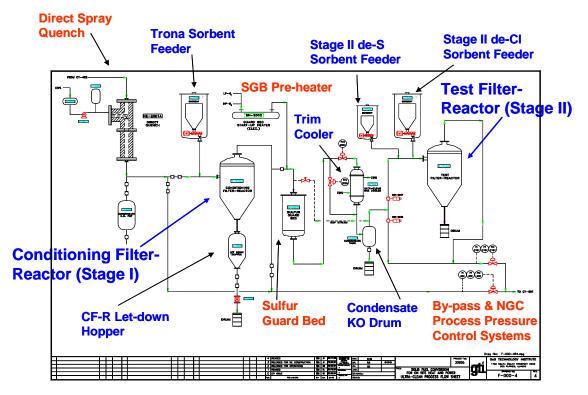


Figure 11 - P&ID for the Ultra-Clean Process Section in the Flex-Fuel Test Facility



Figure 12 – Loading of Candles into Filter Vessel



Figure 13 – Filter Elements (Candles)

In the Conditioning Filter-Reactor, essentially all the sorbent and the entrained solids from the Gasifier are captured on the surface of the filter elements (i.e., candles). Periodically, the solids captured on the filter elements are partially dislodged by pulses of regulated high-pressure nitrogen from a Pulse Control Skid (shared by both Conditioning and Test sections). The timing for these "blow-back" pulses can be determined by build-up of differential pressure across the filter elements, but may also be timed arbitrarily. Captured solids (entrained gasifier material and spent sorbent) flow from the bottom of the Conditioning Filter-Reactor into a Let-Down Hopper. The solids in this collection drum can be isolated from the Filter-Reactor by double block valves, de-pressured to the Vent System, and then unloaded through a Rotary Valve to a drum for post-test analysis and disposal.

The cleaned syngas exits the Conditioning Filter-Reactor from the top section. It can be split -either to the Sulfur Guard Bed, or, under flow control, to bypass the Sulfur Guard Bed, which has been pre-heated to  $> 400^{\circ}$ F to avoid steam condensation and damaging the ZnO material in the SGB vessel. The objective is to provide a pre-determined level of sulfur compounds (1 to 5 ppmv), in the re-mixed syngas stream to the Test section of the NGC Process. Pressure is also reduced from about 285 psig (20.7 bar) to about 215 psig (15.8 bar) for the Testing section.

The re-mixed syngas, with the desired total sulfur and halide concentrations, enters the Syngas Trim Cooler at a reduced pressure of about 230 psig (16.9 bar). The syngas is cooled on the tube side of the cooler from an inlet temperature between 700 and 800°F to an effluent range of 550 to 650°F, as it flows vertically downward. On the shell side of the Syngas Trim Cooler, cooling water flow is controlled to give the desired syngas outlet temperature on the tube side. This stream is relatively clean, with very little (if any) solids loading. The cooled syngas is routed to a Condensate Tank, to allow liquids to drop out. The overhead syngas from the Condensate Tank can then be routed to either the Test Filter-Reactor or through a bypass towards the Tertiary Cyclone and then the Flex-Fuel Flare.

The cooled syngas out of the Syngas Trim Cooler is sent to the lower section of the Test Filter-Reactor. Two sorbents are injected into the conditioned syngas upstream of the Test Filter-Reactor: one for capture of reduced sulfur compounds and one for capture of halides (mostly HCl). The G-72E Stage II sulfur sorbent is metered into the syngas stream from a 1-ft<sup>3</sup> hopper, by a loss-in-weight feeding system enclosed in a pressure vessel (Figure 16). The sulfur sorbent, in the form of very fine powder (~ 20  $\mu$ m average particle size), is injected at rates between 0.04 and 0.3 lb/hr (20-140 grams/hr). The sulfur sorbent is moved into the syngas stream by dilute-phase transport in nitrogen, which picks up the sorbent under the loss-in-weight mechanism. The G-92C Stage II halide sorbent is metered into the syngas stream from a 4-ft<sup>3</sup> hopper by a loss-in-weight feeding system enclosed in a pressure vessel. The halide sorbent, also in a very fine form, is added at injection rates between 0.1 and 2.3 lb/hr (45-1050 grams/hr). Periodically, the sulfur or HCl sorbent addition can be stopped, the feeding vessels de-pressured, the sorbent supply in the hopper replenished, the vessels re-sealed and re-pressured, and sorbent addition re-started.

In the Test Filter-Reactor essentially all the spent sorbents and any residual entrained solids from the Gasifier are captured on the surface of the filter candles. Periodically, these solids are partially dislodged by pulses of regulated high-pressure nitrogen from the Pulse Control Skid. These "blow-back" pulses can be accomplished automatically at timed intervals, or more frequently upon reaching a target differential pressure across the filter elements. Captured solids (mostly spent sorbents) are held in the bottom cone and lower cylinder of the Test Filter-Reactor vessel throughout the duration of a test campaign. At the conclusion of a test series, the Test Filter-Reactor is de-pressured to the Vent System, and the waste solids dropped through double block valves to a drum for analysis and proper disposal. The cleaned syngas exits the Test Filter-Reactor from the top section, flows towards pressure control valves and then the Tertiary Cyclone.

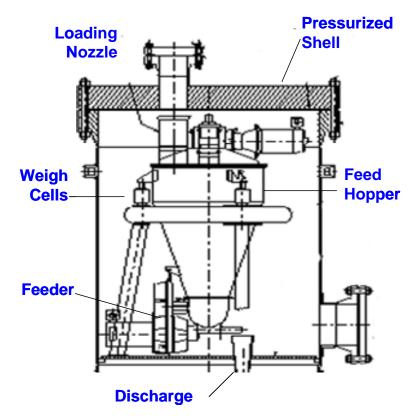


Figure 14- Schematic of Loss-in-Weight Fine Sorbent Feeding System



Figure 15 – Loss-in-Weight Fine Sorbent Feeder



Figure 16 – Stage II Fine Sorbent Feeding System Pressure Vessels

## 7. TEST PLAN

The Base Program identified the probable sorbents and operating conditions for the two stages of the Novel Gas Cleaning Process to achieve very stringent syngas cleaning levels of < 60 ppbv sulfur and < 10 ppbv halide. Table 3 lists the sorbents and operating conditions as well as the estimated, nominal stream flows and pressures for the Conditioning Filter-Reactor and the Test Filter-Reactor that would be applied in the three program test campaigns.

All of the sorbents are commercial, with the possible exception of the two mercury sorbents, which were selected through a comprehensive laboratory test program and interactions with other R&D organizations. Except for trona and nahcolite, which were procured in fine form from the commercial supplier, all of the sorbents are sized (ground and sieved) to -325 mesh at GTI, with an estimated massmean diameter of 20  $\mu$ m. The Stage I and Stage II inlet temperatures are controlled process variables in the program. The syngas pressure is fixed in the test program, and the syngas flow rate is set by the Flex-Fuel gasifier operating capacity. The inlet sulfur and halide contaminant levels are also controlled process variables, but with some uncertainty as to the levels of control that can be achieved in the process. The ranges of sorbent feed rates listed (moles of sorbent active component divided by the moles of the specific contaminant) reflect the expected range that might be required to achieve the targeted gas cleaning performance.

Filter-Reactor	Conditioning	Test
Inlet temperature (°F)	830	550
Inlet pressure (psia)	277	215
Inlet syngas flow (lb/hr)	3439	3648
Filter Face velocity (ft/min)	2.7	3.1
Inlet total sulfur content (ppmv)	not applicable	1-5
Inlet halide content (ppmv)	150 - 500	1-5
Sulfur sorbent (-325 mesh)	none	G-72E
Halide sorbent (-325 mesh)	trona	G-92C
Target sulfur outlet content (ppmv)	not applicable	0.06
Target halide outlet content (ppmv)	1 - 5	0.01
Sulfur and halide sorbent feed rates	2 - 10	2-10
(mole/mole contaminant)		
Mercury sorbents (-325 mesh)	none	GTI-Hg-S9
		TDA's Hg Removal Sorbent
Mercury sorbent feed rate	none	1000 - 2000
(sorbent-to-Hg <sup>0</sup> mass ratio)		

Table 3 - Conditioning Filter-Reactor and Test Filter-Reactor Nominal Test Conditions

## 7.1 Conditioning Filter-Reactor Testing

The Conditioning Filter-Reactor testing objective is to collect data on the ability of the Conditioning Filter-Reactor to achieve the target performance level of 1-5 ppmv halide in the outlet gas and particulate less than 0.1 ppmw. The major test variables are:

- the halide sorbent feed rate, in the range of 2 to 10 molar feed ratio,
- the Conditioning Filter-Reactor inlet temperature, about 443°C (830°F),
- the Conditioning Filter-Reactor pulse cleaning frequency.

Secondary test variables are:

- the filter-reactor face velocity (which may be increased from its nominal value listed in Table 3 by removing selected filter elements),
- the sorbent type (nahcolite instead of trona, for example).

Both of the secondary variables are to be considered only if the filter-reactor does not meet its performance targets within the range of major test variables, or if the performance targets are achieved and additional test time remains.

Conditioning Filter-Reactor testing is the focus of the entire first test campaign, and the Test Filter-Reactor is isolated and not operated during this first test campaign. The consideration of the Conditioning Filter-Reactor is continued by monitoring its fixed operating conditions and performance over the remaining two test campaigns of the program. For purposes of sizing test equipment and identifying sorbent test quantities, it was assumed that each campaign would accumulate a maximum "operation period" of 5 days (120 hours) and a maximum "test time" of 4 days (96 hours).

The planned test matrix is shown in Table 4, and assumes that three full days of controlled test data are gathered. The first campaign would be held at nominal conditions of inlet temperature with variation over the full range of halide sorbent feed rates. The halide sorbent feed rates are shown to be varied initially, on each of the first two test days. On the third test day, the "optimum" halide sorbent feed rate is to be used.

Campaign 1	Day 1	Day 2	Day 3
temperature (°F)	830	830	830
halide sorbent rate (mole/mole)	0	5, 2, 10	optimum
pulse cleaning frequency (1/hr)	1 - 10	1 – 10	1 - 10
Campaign 2-3			
temperature (°F)	830	830	830
halide sorbent rate (mole/mole)	optimum	Optimum	optimum
pulse cleaning frequency (1/hr)	1 - 10	1 – 10	1 - 10

 Table 4 - Conditioning Filter-Reactor Representative Test Matrix

Three day periods (24 hours each) of test running are assumed, with an initial period of each campaign (up to 2 days) being devoted to achieving steady gasifier and conditioning process operation at the desired conditions and confirmation of the controlled variables for the test campaign. The test conditions of subsequent campaigns 2 and 3 would be selected from the results of Test Campaign 1, and the Table 3 listed test conditions are those that would be selected if the process performance targets were achieved during Test Campaign 1. There would be a delay between each of the subsequent test campaigns 2 and 3 of several weeks used to analyze test data, maintain test facility equipment, select the subsequent test run conditions and modify test equipment as required. As stated above, the ability to modify the test results. The subsequent test campaign conditions might also be selected as the expected optimum conditions (as shown in Table 3) and held over several days to confirm steady, reliable behavior.

The key process measurements needed to conduct and monitor the test program are:

- Conditioning Filter-Reactor inlet and outlet syngas mass flow rate,
- Conditioning Filter-Reactor inlet and outlet syngas temperature,
- Conditioning Filter-Reactor halide sorbent mass flow rate,
- Conditioning Filter-Reactor inlet syngas sulfur (H<sub>2</sub>S and COS) and halide (HCl, HBr, etc) contents,
- Conditioning Filter-Reactor outlet syngas sulfur (H<sub>2</sub>S and COS) and halide (HCl, HBr, etc) contents.

Secondary measurements needed to assess the tests are:

- Conditioning Filter-Reactor inlet syngas major constituent composition (H<sub>2</sub>, H<sub>2</sub>O, CH<sub>4</sub>, CO, CO<sub>2</sub>, N<sub>2</sub>),
- Conditioning Filter-Reactor inlet and outlet secondary contaminants (NH<sub>3</sub>, HCN, Hg, particulate),
- Conditioning Filter-Reactor inlet pressure and pressure drop,
- Conditioning Filter-Reactor feed sorbent and collected waste sorbent particle size distributions and compositions,
- Conditioning Filter-Reactor collected waste sorbent accumulated mass.

The pulse cleaning frequency, pulse gas consumption rate, and pressure drop recovery of the Conditioning Filter-Reactor would also be recorded during the testing. Following each campaign, the Conditioning Filter-Reactor would be inspected, cleaned, and repaired as needed. The nature of the filter cakes accumulated on the filter elements, and the accumulation of char and sorbent within the filter-reactor vessels would also be noted.

The methods to be applied to make the process measurements are:

- Conditioning Filter-Reactor syngas mass flow rate: orifice meter continuous readout,
- Conditioning Filter-Reactor inlet and outlet syngas temperature: thermocouple continuous readouts,
- Conditioning Filter-Reactor halide sorbent mass flow rate: feed hopper continuous weight measurement,
- Conditioning Filter-Reactor inlet syngas sulfur (H<sub>2</sub>S and COS) and halide (HCl, HBr, etc) contents: sulfur species by a gas chromatograph equipped with a flame photometric detector; halide species by periodic recovery into deionized water followed by liquid sample injection into an ion chromatograph,
- Conditioning Filter-Reactor outlet syngas sulfur (H<sub>2</sub>S and COS) and halide (HCl, HBr, etc) contents: sulfur species by a gas chromatograph equipped with a flame photometric detector; halide species by periodic recovery into deionized water followed by liquid sample injection into an ion chromatograph,
- Conditioning Filter-Reactor inlet syngas major constituent composition (H<sub>2</sub>, H<sub>2</sub>O, CH<sub>4</sub>, CO, CO<sub>2</sub>, N<sub>2</sub>): water condensed out and periodically weighed; dry gas analyzed by a gas chromatograph equipped with a thermal conductivity detector (TCD),
- Conditioning Filter-Reactor inlet and outlet secondary contaminants (NH<sub>3</sub>, HCN, Hg, particulate): NH<sub>3</sub> by bubbling into an acid solution followed by electrode analysis or a calorimetric method; HCN by bubbling in sodium hydroxide solution followed by a calorimetric method; Hg by an on-line mercury analyzer (PS Analytical Sir Galahad II CEM Analyzer); particulate by periodic isokinetic syngas sampling,
- Conditioning Filter-Reactor inlet pressure and pressure drop: pressure transducer continuous readouts,
- Conditioning Filter-Reactor feed sorbent and collected waste sorbent particle size distributions and compositions: periodic sampling of feed sorbent supply and collected sorbent waste (about once every 24 hours),
- Conditioning Filter-Reactor collected waste sorbent accumulated mass: mass record of each lock hopper drainage.

Of course, during all of the Conditioning Filter-Reactor testing, the conditions and performance of the other components of the process must be monitored and controlled for the testing to be successful: the gasification process, the raw syngas cooler, the sulfur guard bed, the sorbent feed systems, the waste solids handling equipment, and the syngas exhaust treating system.

## 7.2 Test Filter-Reactor Testing

The Test Filter-Reactor testing would collect data on the ability of the Test Filter-Reactor to achieve the target performance levels of 60 ppbv sulfur and 10 ppbv halide in the outlet gas and 90 to 99% mercury removal. The major test variables are:

- the sulfur sorbent feed rate, over the range of 2 to 10 molar feed ratio,
- the halide sorbent feed rate, over the range of 2 to 10 molar feed ratio,
- the stage inlet temperature, over the range of 232 316°C (450 600°F),
- the Test Filter-Reactor pulse cleaning frequency,
- the mercury sorbent type.

Secondary test variables are

- the Test Filter-Reactor face velocity, which may be increased from its nominal value listed in Table 3 by removing selected filter elements,
- the sulfur or halide sorbent type.

Both of the secondary variables would only be considered if the filter-reactor does not meet its performance targets within the range of major test variables, or if the performance targets are achieved and additional test time remains.

Test Filter-Reactor testing would be conducted over the last 2 test campaigns of the threecampaign program. Each test campaign would accumulate a maximum "operation period" of 5 days (120 hours) and a maximum "test time" of 4 days (96 hours). The planned test matrix is shown in Table 5 and assumes three days of controlled test data are gathered in each test campaign. The first campaign (Test Campaign 2) would be devoted to Stage II sulfur and halide removal at representative inlet temperature with variation over the full range of sulfur and halide sorbent feed rates and Test Filter-Reactor pulse cleaning frequency. Test Campaign 3 would operate similarly, but at the lower range of temperatures for mercury removal. Three one-day periods (24 hours each) of testing are assumed, with an initial period of each test campaign (up to 2 days) being devoted to achieving steady gasifier and conditioning process operation at the desired conditions and confirmation of the controlled variables for the test campaign. Specifically, during this initial test period for each test campaign, the halide sorbent (trona) feed rate to the Conditioning Filter-Reactor must be established so that the inlet halide content of the conditioned syngas to the Test Filter-Reactor achieves its target of 1-5 ppmy halide, and the sulfur control achieved in the fixed guard bed must reach steady performance at its target value. The sulfur and halide sorbent feed rates are shown to be varied initially, on each of the first two test days, individually with the other set to zero flow rate. On the third test day, the "optimum" sulfur and halide sorbent feed rates are used.

Campaign 2 - Stage II Sulfur and Halide	Day 1	Day 2	Day 3
temperature (°F)	550	550	550
sulfur sorbent rate (mole/mole)	5, 2, 10	0	optimum
halide sorbent rate (mole/mole)	0	5, 2, 10	optimum
pulse cleaning period (hr)	5 - 20	5 - 20	5 - 20
Campaign 3 - Mercury			

 Table 5 - Test Filter-Reactor Representative Test Matrix

temperature (°F)	300-450	300-450	300-450
mercury sorbent	1	2	best
mercury sorbent rate (lb/lb)	1000-2000	1000-2000	optimum
pulse cleaning period (hr)	5 - 50	5 - 50	5 - 50

There would be a delay between Test Campaigns 2 and 3 of several weeks used to analyze test data, maintain test facility equipment, select the subsequent test run conditions and modify test equipment as required. As stated above, the ability to modify the test sorbent type and/or the filter-reactor face velocity is also a test option if dictated by the previous test results.

The key process measurements needed to conduct and monitor the test program are:

- Test Filter-Reactor inlet and outlet syngas mass flow rate,
- Test Filter-Reactor inlet and outlet syngas temperature,
- Test Filter-Reactor sulfur sorbent mass flow rate,
- Test Filter-Reactor halide sorbent mass flow rate,
- Test Filter-Reactor inlet syngas sulfur (H<sub>2</sub>S and COS) and halide (HCl, HBr, etc) contents,
- Test Filter-Reactor outlet syngas sulfur (H<sub>2</sub>S and COS) and halide (HCl, HBr, etc) contents. Secondary measurements needed to assess the tests are:
- Test Filter-Reactor inlet syngas major constituent composition (H2, H2O, CH4, CO, CO2, N2),
- Test Filter-Reactor inlet and outlet secondary contaminants (NH3, HCN, Hg, particulate),
- Test Filter-Reactor inlet pressure and Conditioning Filter-Reactor pressure drop,
- Test Filter-Reactor feed sorbent and collected waste sorbent particle size distributions and compositions,
- Test Filter-Reactor collected waste sorbent accumulated mass,
- The Conditioning Filter-Reactor conditions: halide sorbent (trona) feed rate, inlet and outlet temperature.

The pulse cleaning frequency, pulse gas consumption rate, and pressure drop recovery of the Test Filter-Reactor would be recorded during the testing. Following each test campaign, the Test Filter-Reactor would be inspected, cleaned, and repaired as needed. The nature of the filter cakes accumulated on the filter elements, and the accumulation of char and sorbent within the filter-reactor vessels would also be noted.

The methods to be applied to make the process measurements are:

- Test Filter-Reactor syngas mass flow rate: orifice meter continuous readout,
- Test Filter-Reactor inlet and outlet syngas temperature: thermocouple continuous readouts,
- Test Filter-Reactor sulfur sorbent mass flow rate: feed hopper continuous weight measurement,
- Test Filter-Reactor halide sorbent mass flow rate: feed hopper continuous weight measurement,
- Test Filter-Reactor inlet syngas sulfur (H<sub>2</sub>S and COS) and halide (HCl, HBr, etc) contents: sulfur species by a gas chromatograph equipped with a flame photometric detector; halide species by periodic recovery into deionized water followed by liquid sample injection into an ion chromatograph,
- Test Filter-Reactor outlet syngas sulfur (H<sub>2</sub>S and COS) and halide (HCl, HBr, etc) contents: sulfur species by a gas chromatography technique developed in Base Program (based on an ASTM procedure); halide species by continuous recovery into deionized water followed by continuous ion chromatography analysis,

- Test Filter-Reactor inlet syngas major constituent composition (H<sub>2</sub>, H<sub>2</sub>O, CH<sub>4</sub>, CO, CO<sub>2</sub>, N2): water condensed out and periodically weighed; dry gas analyzed by a gas chromatograph equipped with a thermal conductivity detector (TCD),
- Test Filter-Reactor inlet and outlet secondary contaminants (NH<sub>3</sub>, HCN, Hg, particulate): NH<sub>3</sub> by bubbling into an acid solution followed by electrode analysis or a calorimetric method; HCN by bubbling in sodium hydroxide solution followed by a calorimetric method; Hg by an on-line mercury analyzer (PS Analytical Sir Galahad II CEM Analyzer); particulate by periodic isokinetic syngas sampling,
- Test Filter-Reactor inlet pressure and Conditioning Filter-Reactor pressure drop: pressure transducer continuous readouts,
- Test Filter-Reactor feed sorbent and collected waste sorbent particle size distributions and compositions: periodic sampling of feed sorbent supply and collected sorbent waste (once every 24 hours),
- Test Filter-Reactor collected waste sorbent accumulated mass: mass record of each lock hopper drainage.

Of course, during all of the Test Filter-Reactor testing, the conditions and performance of the other components of the process must be monitored and controlled for the testing to be successful: the gasification process, the raw syngas cooler, the Conditioning Filter-Reactor, the Sulfur Guard Bed, the second Indirect Syngas Cooler (Trim Cooler), the sorbent feed systems, the waste solids handling equipment, and the syngas exhaust treating system.

Further details on the scope of each testing campaign are provided in Table 6, Table 7, and Table 8. As can be seen in these tables, testing durations were estimated at 72 hours of continuous testing for the first test campaign and at 88 hours for each of the second and third test campaigns. These durations were estimated based on reasonable estimates of durations for the various test segments (or set points) to achieve program goals. The analytical scope of work was designed to meet program goals and includes "semi-continuous" measurements with "on-line" analytical instruments as well as time-integrated (batch) samples to be analyzed by GTI's Chemical Services Research group to confirm "on-line" measurements and determine the concentrations of additional compounds of interest to the program.

The various process samples (defined in the following section of this report) include fuel feedstock, ash, raw syngas, fresh sorbent materials, spent sorbents, condensed liquids, and gas samples throughout the NGC Process section. These samples would be extracted using especially designed sampling and conditioning systems. These systems and the necessary analytical instruments and procedures are discussed in detail in the following report section.

Table 6 – First Test Campaign: Evaluate Performance of Conditionin	g Filter-Reactor (CF-R) for Combined Particulate/Halide Removal
Tuble o The Test Compargne Litanaate Terrormanee of Conardonn	g i neer recueetor (or re) for compined i di ticulate, riande recino (di

Test Segment or Activity	Estimated Duration, hrs	Description
Gasifier start up & heating up of NGC Process conditioning section (CF-R product gas going directly to Flare Gas Cyclone (FGC))	24	
CF-R testing under first set point (SP#1) SS conditions	8	(no trona injection, 830F, 1-10 pulse cleaning frequency)
CF-R testing under second set point (SP#2) SS conditions	8	(inject trona at Na/CI=5, 830F, 1-10 pulse cleaning frequency)
CF-R testing under third set point (SP#3) SS conditions	8	(inject trona at Na/Cl=2, 830F, 1-10 pulse cleaning frequency)
CF-R testing under fourth set point (SP#4) SS conditions	8	(inject trona at Na/Cl=10, 830F, 1-10 pulse cleaning frequency)
CF-R testing under fifth set point (SP#5) SS conditions	8	(inject trona at Na/Cl=optimum, 830F, 1-10 pulse cleaning frequency)
Shutdown	8	
	72	

#### ANALYTICAL WORK

Sampling Point	Purpose	No. of Batch Samples to be Taken	No. of Batch Samples to be Analyzed
G-7	Determine efficiency of primary & secondary cyclones	15	5
G-8	Determine complete syngas composition and concentrations of other	15	5
	species of interest:		
	Bulk: H2, H2O, CH4, CO, CO2, N2		
	Minor: H2S, COS, HCl, particulate		
	Other species of interest: TBD		
G-13	Confirm semi-continuous HCI and particulate measurements	15	5
	in the CF-R product gas & determine fate of other species of interest	-	
S-12	Confirm HCI removal & characterize particulate/spent trona waste	15	5
	for disposal		
?	To satisfy environmental emissions requirements?		
S-1	Feedstock: How uniform is selected carbonaceous feedstock?	15	5 (may or may not be needed)
S-5	Gasifier ash: Characterize to satisfy disposal requirements	15	5
S-6	Dust from Secondary Cyclone: Charaterize to satisfy disposal requirements	15	5
S-21	Dust from Flare Gas Cyclone: Charaterize to satisfy disposal requirements	15	5
S-7C	Primary Cyclone return	15	5
S-7D	Bottom fluid-bed	15	5
S-7E	Middle fluid-bed	15	5
S-7F	Top fluid-bed	15	5

# Table 7 – Second Test Campaign: Evaluate Performance of Test Filter-Reactor (TF-R) for Sulfur and Halide Removal (Separately and Together)

Note: Assumes CF-R is operating under optimum operating conditions (830°F, 1-10 pulse cleaning frequency, and trona injected at Na/Cl optimum)

Test Segment or Activity	Estimated Duration, hrs	
Gasifier start up & heating up of CF-R & Sulfur Guard Bed (SGB) in NGC Process Conditioning section	24	
Establishment of conditioned syngas conditions & warming up of Test Filter-Reactor (TF-R)	8	
TF-R testing under first set point (SP#1) SS conditions	6	(sulfur sorbent injected at Zn/S=5, no halide sorbent injection, 550F, 5-20 pulse cleaning frequency)
TF-R testing under second set point (SP#2) SS conditions	6	(sulfur sorbent injected at Zn/S=2, no halide sorbent injection, 550F, 5-20 pulse cleaning frequency)
TF-R testing under third set point (SP#3) SS conditions	6	(sulfur sorbent injected at Zn/S=10, no halide sorbent injection, 550F, 5-20 pulse cleaning frequency)
TF-R testing under fourth set point (SP#4) SS conditions	6	(no sulfur sorbent injection, halide sorbent injected at Na/Cl=5, 550F, 5-20 pulse cleaning frequency)
TF-R testing under fifth set point (SP#5) SS conditions	6	(no sulfur sorbent injection, halide sorbent injected at Na/CI=2, 550F, 5-20 pulse cleaning frequency)
TF-R testing under sixth set point (SP#6) SS conditions	6	(no sulfur sorbent injection, halide sorbent injected at Na/Cl=10, 550F, 5-20 pulse cleaning frequency)
TF-R testing under seventh set point (SP#7) SS conditions	12	(sulfur sorbent injected at Zn/S=optimum, halide sorbent injected at Na/Cl=optimum, 550F, 5-20 pulse cleaning frequency)
Shutdown	8	
	88	

#### ANALYTICAL WORK

Guidelines: No need to take batch samples up to and including G-13 (rely on semi-continuous measurements of species of direct interest to CF-R; or those that have impact on TF-R) OR, take samples and store them, in case it becomes necessary to do some extra analyses for additional insight

Sampling Point	Purpose	No. of Batch Samples to be Taken	Number of Batch Samples to be Analyzed
G-14A	Assess desulfurization performance of SGB		
G-14B	Determine complete composition (bulk, minor, trace) of conditioned syngas	3	1
	(look particularly for COS since fate across SGB is not known)		
	(conditioned syngas going directly to Flare Gas Cyclone (FGC))		
G-14	Confirm semi-continuous S, HCI, and particulate in the TF-R inlet syngas	21	7
	& determine overall composition and concentrations of other species of interest		
G-19	Confirm semi-continuous S, HCl, and particulate in the TF-R product gas	21	7
	& determine overall composition and concentrations of other species of interest		
S-19	Confirm S and/or HCI removal & characterize particulate/spent sorbents waste for	21	7
	disposal		
?	To satisfy environmental requirements?		
S-1	Feedstock: How uniform is selected carbonaceous feedstock?	21	7 (may or may not be needed)
S-5	Gasifier ash: Characterize to satisfy disposal requirements	21	7
S-6	Dust from Secondary Cyclone: Charaterize to satisfy disposal requirements	21	7
S-21	Dust from Flare Gas Cyclone: Charaterize to satisfy disposal requirements	21	7
S-7C	Primary Cyclone return	21	7
S-7D	Bottom fluid-bed	21	7
S-7E	Middle fluid-bed	21	7
S-7F	Top fluid-bed	21	7

# Table 8 – Third Test Campaign: Evaluate Performance of Test Filter-Reactor (TF-R) for Mercury Removal (no Stage II Sulfur or Halide Sorbent to be Injected)

Note: Assumes CF-R is operating under optimum operating conditions (830F, 1-10 pulse cleaning frequency, and trona injected at Na/Cl optimum)

Test Segment or Activity	Estimated Duration, hrs	
Gasifier start up & heating up of CF-R & Sulfur Guard Bed (SGB) in NGC Process Conditioning section	24	
Establishment of conditioned syngas conditions & warming up of Test Filter-Reactor (TF-R)	8	
TF-R testing under first set point (SP#1) SS conditions	6	(lower-T mercury sorbent injected at sorbent/Hg=1000, 300F, 5-50 pulse cleaning frequency)
TF-R testing under second set point (SP#2) SS conditions	6	(lower-T mercury sorbent injected at sorbent/Hg=1500, 300F, 5-50 pulse cleaning frequency)
TF-R testing under third set point (SP#3) SS conditions	6	(lower-T mercury sorbent injected at sorbent/Hg=2000, 300F, 5-50 pulse cleaning frequency)
Load higher-T mercury sorbent	6	
TF-R testing under fourth set point (SP#4) SS conditions	6	(higher-T mercury sorbent injected at sorbent/Hg=1000, 450F, 5-50 pulse cleaning frequency)
TF-R testing under fifth set point (SP#5) SS conditions	6	(higher-T mercury sorbent injected at sorbent/Hg=1500, 450F, 5-50 pulse cleaning frequency)
TF-R testing under sixth set point (SP#6) SS conditions	6	(higher-T mercury sorbent injected at sorbent/Hg=2000, 450F, 5-50 pulse cleaning frequency)
TF-R testing under seventh set point (SP#7) SS conditions	6	(better mercury sorbent injected at sorbent/Hg=optimum, 300 or 450F, 5-50 pulse cleaning frequency)
Shutdown	8	
	88	

#### ANALYTICAL WORK

Guidelines: No need to take batch samples up to and including G-13 (rely on semi-continuous measurements of species of direct interest to CF-R; or those that have impact on TF-R) OR, take samples and store them, in case it becomes necessary to do some extra analyses for additional insight.

Put less emphasis on establishing S and HCl levels in conditioned syngas, and more of our resources on Hg analyses

do complete solids and liquids analyses for Hg to perform mass balance in the entire system

Sampling Point	Purpose	No. of Batch Samples to be Taken	No. of Batch Samples to be Analyzed
G-14A	Assess desulfurization performance of SGB		
G-14B	Determine complete composition (bulk, minor, trace) of conditioned syngas	3	1
	(look particularly for COS since fate across SGB is not known)		
	(conditioned syngas going directly to Flare Gas Cyclone (FGC))		
G-14	Confirm semi-continuous S, HCI, Hg, and particulate in the TF-R inlet syngas	21	7
	& determine overall composition and concentrations of other species of interest		
G-19	Confirm semi-continuous S, HCI, Hg, and particulate in the TF-R product gas	21	7
	& determine overall composition and concentrations of other species of interest		_
S-19	Confirm Hg removal & characterize particulate/spent sorbent waste for	21	7
	disposal		
?	To satisfy environmental requirements?		
S-1	Feedstock: How uniform is selected carbonaceous feedstock?	21	7 (may or may not be needed)
S-5	Gasifier ash: Characterize to satisfy disposal requirements	21	7
S-6	Dust from Secondary Cyclone: Charaterize to satisfy disposal requirements	21	7
S-21	Dust from Flare Gas Cyclone: Charaterize to satisfy disposal requirements	21	7
S-7C	Primary Cyclone return	21	7
S-7D	Bottom fluid-bed	21	7
S-7E	Middle fluid-bed	21	7
S-7F	Top fluid-bed	21	7

## 8. SAMPLE EXTRACTION & CONDITIONING SYSTEMS AND ANALYTICAL INSTRUMENTATION

An important objective of the Novel Gas Cleaning Optional Program is to verify, at the pilotscale, the laboratory test results obtained in the Base Program with respect to operating temperatures for Stages I and II of the NGC Process, sorbent selections, sorbent/contaminant ratios, and ability of the process to accomplish very stringent contaminant levels in the syngas (i.e., particulates < 0.1 ppmw, total reduced sulfur compounds ( $H_2S$  and COS) < 60 ppby, total halides (mainly HCl) < 10 ppby, and mercury (mainly elemental mercury,  $Hg^0$ ) < 0.01 ppby. To accomplish these goals, GTI, in collaboration with Siemens, developed a sampling and analysis protocol, as part of the Test Plan for the program, which defined the sampling locations, sample type (solid, gas, or liquid), sample description, purpose, number of samples, analyses to be performed, and measurement methods, during each NGC program test campaign. Although the protocol was established as a general guideline that could be modified as needed based on the specific requirements for each test campaign, every effort was made to ensure that the scope of work allowed for development of the necessary data to accomplish Optional Program goals, that the state of the raw syngas was well defined before and after conditioning, and most importantly that performance of Stage II of the process could be properly assessed. Moreover, sufficient data would be collected to develop adequate material and energy balances for the gasifier to assess its efficiency in generating syngas from the test feedstock. The data generated would also permit characterization of process emissions for permitting activities and gasification and syngas cleanup byproduct materials for assessment of disposal options. As shown in Figure 17, process samples included fuel feedstock, ash, raw syngas, fresh sorbent materials, spent sorbents, and condensed liquids, in addition to several gas samples throughout the NGC Process section.

A list of the samples and their designations (solid = S, gas = G, liquid = L) is presented below. These sample locations are identified in Figure 17. Points listed without a S, G, or L designation are flow streams that can be calculated based on material balance considerations, but are not sampled. For each sampling point relevant to the NGC Process testing program, Table 9 provides temperature, pressure, and expected (estimated) concentrations of bulk gas constituents (i.e., H<sub>2</sub>, CH<sub>4</sub>, CO, CO<sub>2</sub>, H<sub>2</sub>O, N<sub>2</sub>), major and minor contaminants (particulates, total sulfur (H<sub>2</sub>S, COS), total halides (HCl, HBr), ammonia (NH<sub>3</sub>), HCN), and trace contaminants (Hg).

Solid Samples: feedstocks, gasifier ash, gas particulates, fresh sorbents, spent sorbents

- S-1 Carbonaceous feedstock (Bethlehem Coke Breeze, washed Indian coal, North Dakota lignite)
- S-5 Gasifier ash
- S-6 Dust from Secondary Cyclone
- S-7C Primary Cyclone dust recycled to Gasifier
- S-9 Halide Sorbent (Trona) to Conditioning Filter-Reactor (CF-R)
- S-12 Particulates/Spent Halide Sorbent (Trona) mix from CF-R
- S-15A UCP Stage II Sulfur Sorbent (Süd-Chemie's G-72E, finely ground) to Test Filter-Reactor (TF-R)
- S-15B UCP Stage II Halide Sorbent (Süd -Chemie's G-92C, finely ground) to TF-R
- S-18 Spent UCP Stage II Sulfur and Halide Sorbents from TF-R (plus some particulates
- S-21 Flare Cyclone dust
- S-25 Sulfur Guard Bed Sorbent Material (Süd-Chemie's G-72E Catalyst, pellet form)
- S-7D Gasifier Lower Bed Sample
- S-7E Gasifier Middle Bed Sample
- S-7F Gasifier Upper Bed Sample

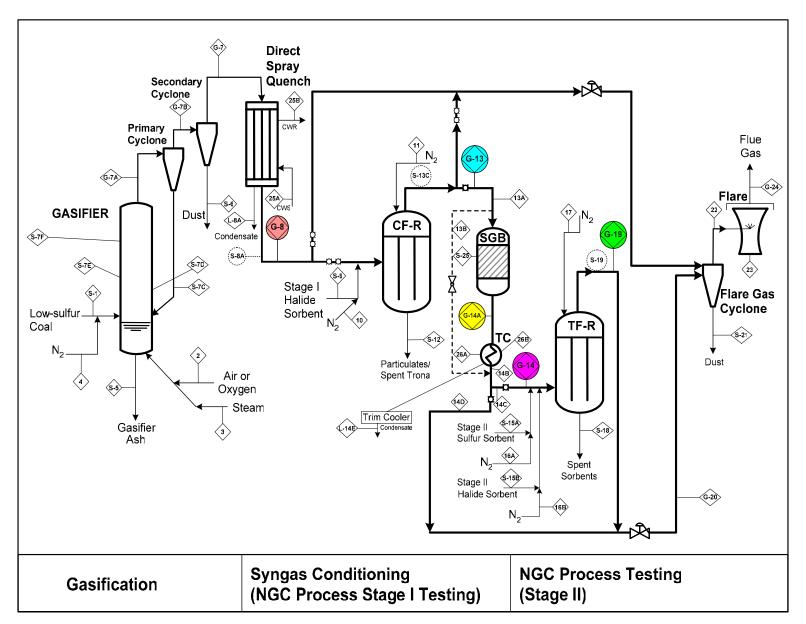


Figure 17 - Ultra-Clean Process Layout Showing Sampling Points for Solids, Liquids, and Gases

Gas Samples: Raw syngas and treated syngas at various stages in the UCP section

- G-7A Raw Syngas Exiting Gasifier
- G-7B Raw Syngas Exiting Primary Cyclone
- G-7 Raw Syngas Exiting Secondary Cyclone
- G-8 Partially-cooled raw syngas exiting Direct Spray Water Quench (DSQ) system
- G-13 Partially-conditioned CF-R product syngas (essentially dust-free, dechlorinated to 1-5 ppmv HCl, but still containing all the raw syngas sulfur)
- G-14A Cooled de-Cl and de-S (desulfurized) Syngas from SGB (before blending with SGB Bypass)
- G-14 Conditioned Syngas (de-Cl, de-S, T, P per UCP specifications) to TF-R: Cooled de-Cl and de-S Syngas from SGB and Trim Cooler (TC) (after blending with SGB By-pass and further cooling if necessary)
- G-19 Ultra-Cleaned Syngas from TF-R
- G-20 Depressurized, Cooled Raw Syngas or Ultra-Cleaned Syngas to Flare Cyclone
- G-24 Flue Gas from Flare

#### Liquid Samples:

- L-8A Condensate from Direct Spray Quench system
- L-14E Condensate from Trim Cooler

Prior to this process demonstration unit (PDU) test program, existing analytical capability for the Flex-Fuel Test Facility included a double-train sample extraction and conditioning (SE&C) system (Figure 18, Figure 19), placed immediately after the Direct Spray Water Quench (i.e., at the G-8 sampling location) and a Rosemount CAT 200 on-line analyzer. As shown in Figure 19, in this conventional system partially-cooled raw syngas passes through a sintered metal filter (Mott<sup>™</sup>) while maintained at elevated temperature to remove particulate matter. Subsequently, particulate free sample gas is cooled and condensate removed. In the presence of condensable hydrocarbon vapors (tars and oils) this is best accomplished by rapid cooling to temperatures below the dew point of all significant condensable species in a configuration that drops them out in knockout pots. The SE&C system shown in Figure 18 uses water to quickly cool the sample gas to about 80°F followed by a second stage of cooling by expansion of high pressure nitrogen. Downstream of the knockout pots, traps, and coalescing filters, sample gas cylinders can then be readily filled as needed and clean sample gas is available for analyzers after pressure letdown.

The CAT 200 Rosemont online analyzer monitors carbon monoxide (CO), oxygen ( $O_2$ ), methane (CH<sub>4</sub>), and hydrogen (H<sub>2</sub>) in the clean (particulate-free), dry syngas sample. It measures CH<sub>4</sub> and CO by NDIR,  $O_2$  by a paramagnetic technique, and H<sub>2</sub> by a thermal conductivity detector (TCD). The analyzer was installed at the FFTF in late April 2004, and its performance is verified regularly by comparing output values to known standards and independent analyses on "grab" samples by GTI's in-house analytical laboratory. The CAT 200 analyzer was also connected to the DCS, allowing the instrument to be monitored from the FFTF Control Room.

The original plan for the analytical work in the NGC Process PDU tests was to install the required instruments (gas chromatographs, mercury analyzers, etc.) and impinger sets to obtain time-integrated (batch) samples at key points in the process (i.e., G-8, G-13, G-14, and G-19). At G-8, for example, these can be installed in parallel with the existing CAT 200 Rosemount analyzer for CO,  $O_2$ , CH<sub>4</sub>, and H<sub>2</sub>. However, accurate measurement of very low levels of sulfur and halide compounds, such as expected especially at G-19, requires the use of specially-prepared and inerted equipment, with Sulfinert<sup>TM</sup> or Silcosteel<sup>TM</sup> tubing and specialized fittings to minimize attachment of low levels of these compounds to surfaces.

Sampling Point	G-7	G-8	G-13	G-14A	G-14	G-19
Temperature, °F	1634	830	800	800	550	533
Pressure, psia	285	285	235	235	215	215
Bulk Gas Constituents, vol% <sup>a</sup>						
$H_2$	13.77	13.77	13.77	13.77	13.77	13.77
CH <sub>4</sub>	2.20	2.20	2.20	2.20	2.20	2.20
СО	15.67	15.67	15.67	15.67	15.67	15.67
CO <sub>2</sub>	10.48	10.48	10.48	10.48	10.48	10.48
H <sub>2</sub> O	12.17	12.17	12.17	12.17	12.17	12.17
N <sub>2</sub>	45.50	45.50	45.50	45.50	45.50	45.50
Major & Minor Contaminants						
Particulates	21,250 <sup>b</sup>	21,250	~ 0	~ 0	~ 0	~ 0
$H_2S + COS (ppmv)$	941 <sup>c</sup>	941	941	< 10 <sup>d</sup>	1-5	0.060
HCl + HBr (ppmv)	100-500 <sup>e</sup>	100-500	1-5	1-5	1-5	0.010
NH <sub>3</sub> + HCN (ppmv)	1,240	1,240	TBD <sup>f</sup>	TBD	TBD	TBD
Trace Contaminants						
Hg (ppbv)	2 <sup>g</sup>	TBD	TBD	TBD	TBD	TBD

 Table 9 - Estimated Temperatures, Pressures, and Contaminant Concentrations at Various

 Sampling Points in the Novel Gas Cleaning Process Section

The NGC PDU test program benefited greatly from a separate ongoing development effort at GTI for novel sample gas conditioning procedures that avoid condensation of species in the process gas. Part of the scope of the "Biomass Gasification Research" projects is development of a state of the art analytical suite to monitor syngas composition.<sup>11,12</sup> This new approach, as noted in Figure 18 and Figure 19 by the "Hot FT-IR Sample Line" bypasses the condensers of traditional gas sample extraction and conditioning systems and provides hot/warm samples directly to analyzers. This is described in more detail below.

<sup>a</sup> Based on air-blown gasification (with no in-situ desulfurization) of a Montana coal (0.62 wt% S and 8.3% ash on a dry basis)

<sup>b</sup> 21,250 ppmw char (nominal GTI expected) – 31,250 ppmw (maximum expected)

<sup>c</sup> Total sulfur (CS<sub>2</sub> ignored); assuming  $[H_2S] \approx 10[COS]$ , about 855 ppmv  $H_2S$  and 86 ppmv COS can be expected

<sup>d</sup> Assuming > 99% sulfur removal by Sulfur Guard Bed (actual fate of COS in SGB not known for certain)

<sup>e</sup> Total halides (*estimate*); only HCl has been taken into consideration in the experimental work.

<sup>f</sup> To be determined for general interest only; filter-reactors may have catalytic activity for NH<sub>3</sub> destruction

<sup>g</sup> Estimate ( 2 ppbv)



Figure 18 - Main FFTF Sample Extraction and Conditioning Skid (Untreated Syngas)

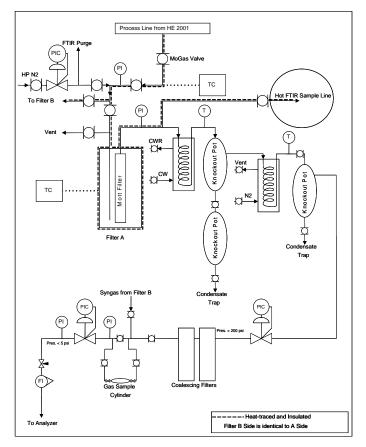


Figure 19 - Sample Conditioning System for Inlet of the Conditioning Filter-Reactor

To meet the requirements of the analytical work scope in the Optional Program, two additional single-train sample extraction and conditioning (SE&C) skids were designed and installed at sampling locations "G-13" (Conditioning Filter-Reactor product gas) and/or "G-14" (Conditioned syngas, inlet to Test Filter-Reactor) and at "G-19" (Test Filter-Reactor product gas – ultra-cleaned syngas). The two additional SE&C skids were installed in the FFTF during early March 2005. Figure 17 shows the process configuration on which the sampling interface systems were installed. In this figure, the gas sampling points are identified and the locations of sample extraction interfaces are highlighted (i.e., G-8, G-13, G-14, G-14A, and G-19). A Varian gas chromatograph system equipped with a pulsed flame photometric detector (PFPD), CP-Cil5 capillary columns, and a cryo-trap, was selected and procured as a suitable instrument for sulfur measurement at the ppbv level. This Trace Sulfur System (TSS) features a built-in high performance sample concentrator, a sensitive PFPD detector with a detection limit of 30 to 50 parts-per-trillion (ppt), and significant capability for speciated and total sulfur analyses. To measure HCl at the ppmv and ppbv levels, glass impingers were setup for chlorine sampling according to EPA Method 26, for subsequent analysis using an ion chromatograph instrument.

The three gas SE&C systems implemented for the PDU test program use the controlled condensation management approach, which incorporates filtration, gas cooling, condensate collection and removal, and drying. The controlled condensation management approach is depicted in Figure 22 (and previously in Figure 19). This process interface provides for separation of suspended particles, condensates, and gases. Particles and condensates are accumulated in the skid for post-test recovery and analysis. The gases, before and after drying stages, can be collected in sample canisters, passed through collection impingers (i.e., for HCl sampling) or absorption traps (i.e., carbon traps for mercury sampling, for example), or sent directly to analytical instruments for on-line measurements. Figure 21 shows a photograph of the controlled condensation SE&C Skid #2 (based on the design shown in Figure 20) for process gas samples from locations G-13, G-14, and G-14A. This photograph shows the system before heat tracing and insulation were completed. Figure 22 shows the same system after heat tracing and insulation were completed.

Two analytical stations were set up in two areas of the FFTF outside of the process area boundary because of Class I, Division II code restrictions on equipment in areas with potentially flammable, explosive gases. Sample lines of from 30 to 55 ft were needed from the various process interface points to accommodate this restriction. All sample lines were heat-traced and insulated to preserve gas sample integrity and to maintain gas temperatures above the instrument inlet conditions. Figure 23 shows the arrangement of components for the sample transport lines for the on-line analyses of gas composition.

A photo of the first analytical station, set up on the second floor of the FFTF, is shown in Figure 24. It includes four instruments:

- an Industrial Machine and Control Corporation (IMACC) Fourier-Transform Infrared Spectrometer (FT-IR)
- a Stanford Research Systems QMS300 Mass Spectrometer (MS)
- an Agilent 5890 gas chromatograph with flame photometric detector (GC/FPD)
- a Varian micro gas chromatograph with thermal conductivity detector (µGC)

The gas sampling system was built to allow for the selection of samples from any of three sources from the process (i.e., G-8, G-13, and G-14/G-14A) and direct the gas stream to one of the above four analyzers. In addition to supplying the on-line analyzers with representative syngas samples, the system shown in Figure 24 was used to extract sample gases to EPA reference method 26 impingers, allowing for direct comparison of batch and FT-IR HCl measurements. The sample network is illustrated in Figure 25. All lines are heat traced to maintain gas temperatures above the instrument inlet conditions.

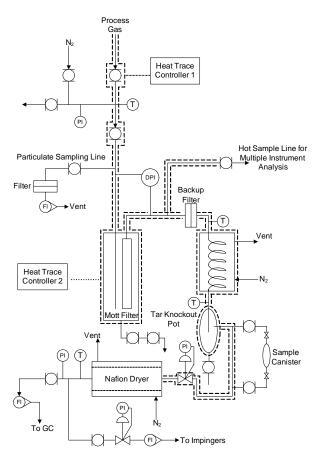


Figure 20 – Ultra-Clean Process Section Second Sample Extraction & Conditioning Skid (Placed Between Conditioning and Test Barrier Filter-Reactors)



Figure 21 - Photograph of the SE&C Skid #2 at the G-13/G-14 Locations (Before Insulation)



Figure 22 - Photograph of the SE&C Skid #2 at the G-13/G-14 Locations (After Insulation)

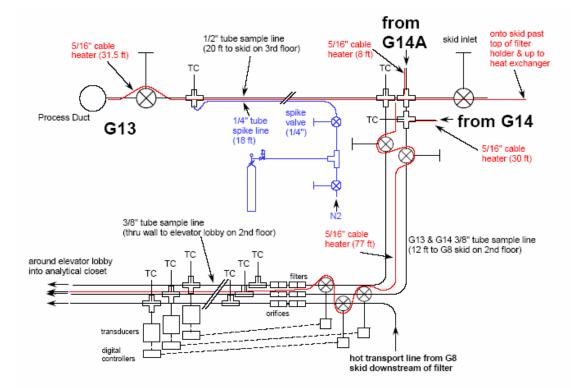


Figure 23 - Gas Sample Transport for On-line Analysis for Ultra-Clean Process in Flex-Fuel Test Facility



Figure 24 - Analytical Instruments Installed in the Second-Floor Gas Analysis Station in the Flex-Fuel Test Facility.

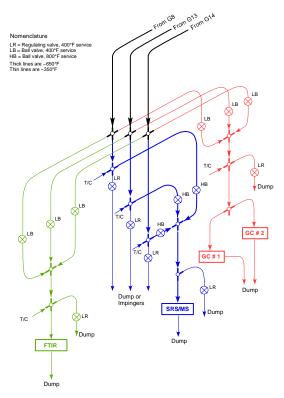


Figure 25 - Sample Switching Network for Multiple Analyzers

The sample transport lines from the raw syngas sample (G-8, before removal of suspended ash particles) used either of the two filters mounted on the main sample extraction & conditioning skid (i.e., SE&C Skid #1) at the G-8 location. The connection to this system is illustrated in Figure 26.

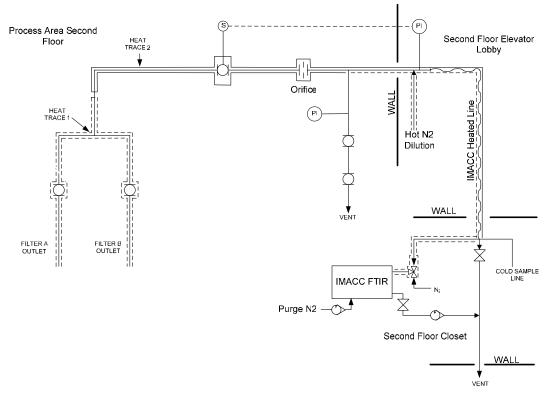


Figure 26 - Illustration of High-Temperature Process Interface for FT-IR Line at G-8.

A photograph of the second analytical station is shown in Figure 27. Close up views of the GC/PFPD installed at the station are presented in Figure 28. This station was set up on the fourth floor of the FFTF in order to meet the analytical work scope requirements of the second test campaign during which critical measurements were made to assess the NGC Process Stage II performance for removal of multiple contaminants (H<sub>2</sub>S, COS, HCl) to ppbv levels, and those of the third test campaign during which mercury removal via dry sorbent injection upstream of the Test Filter-Reactor was investigated. A much simpler flow distribution arrangement was required for this station compared to the second floor gas analysis station. Because the only source of syngas being delivered to this station was from sampling location G-19 (i.e., Test Filter-Reactor product gas), no provisions for syngas source switching had to be made. Therefore at this analysis station, tees were installed in the sample line for sample to be withdrawn for impinger measurements, mass spectrometer measurements, and GC/PFPD measurements. Each of these analyzer streams included its own tee and regulating valve for an adjustable dump line to an outside vent. In addition, the excess flow through the line not entering any of the analyzer streams was also dumped to an outside vent. Although provision was made to install the SRS MS at the fourth floor station, the MS was used exclusively at the second floor station during the NGC Program PDU tests.



Figure 27 – Flex-Fuel Test Facility Fourth Floor Analytical Station



Figure 28 – Trace Sulfur Analysis GC/PFPD Installed at the FFTF Fourth Floor Analytical Station

Except for vent lines, all of the sample lines at each analysis station were heat traced and insulated. Silimar to the lines from G-8, G-13, and G-14/G-14A, the sample transport line from G-19 to the fourth floor analysis station included a manual and actuated ball valve, an in-line filter and orifice, and a pressure transducer to actuate the ball valve in the event excess pressure was detected within the sample line. A cable heater was used to heat trace this line, and the line was insulated. The initial portions of the G-19 sampling line are shown in Figure 29, before heat tracing and insulation were applied.

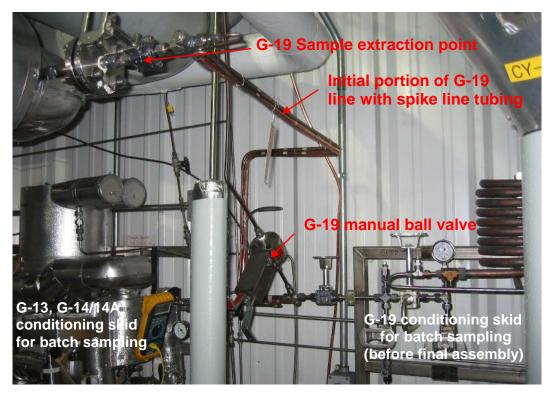


Figure 29 - Initial Sections of the G-19 Sample Location and Sampling Line

The sampling systems were installed to measure reduced sulfur (H<sub>2</sub>S and COS) and halide (HCl) compounds at various concentrations throughout the NGC Process section, ranging from hundreds of parts-per-million (ppmv) to very low concentrations at the parts-per-billion (ppbv) level, as indicated earlier in Table 9. To provide very low-level detection, it was necessary to take extreme care in the selection of construction materials for sampling to avoid contamination and loss of analyte. The construction of an ultimate contamination- and corrosion-free sampling system was achieved with the use of clean materials with minimal active surfaces and suitable passivation. The product selected for heated and pressurized sampling lines was Silcosteel®-CR (Restek Corporation, Bellefonte, PA). Components of the SE&C skids at the G-13 and G-19 locations (tubing, fittings, valves, flow metering orifices, and Mott filters) were coated with chemically inert Silcosteel® and Sulfinert® (also from Restek Corporation) coatings to minimize reactions and adsorption of gases on the surfaces. Silcosteel®-CR was also used for the G-13, G-14, and G-19 sampling lines. The only exception was the G-8 sampling lines which were not coated. Sample canisters and valves were made from Sulfinert® materials. Low pressure and non-heated sampling lines and vessels were constructed of Teflon or borosilicate glass. Flexible silicone tubing was used to make low pressure connections between the coated steel or Teflon tubing and glass apparatus.

The Silcosteel®-CR treatment was developed specifically to protect equipment exposed to hydrochloric acid, nitric acid, sulfuric acid, or marine environments. A Silcosteel®-CR treatment

upgrades the corrosion resistance of 300-grade stainless steels by greater than an order of magnitude. The Sulfinert® coating was developed for inertness to sulfur species. Both coating processes bond a very thin, flexible layer of glass to the stainless steel and prevent direct gas-metal interaction.

### 8.1 Hydrogen Chloride (HCl) Analysis

Hydrogen chloride (HCl) in a gaseous stream is measured by sparging raw or treated syngas streams through glass bubblers containing dilute sulfuric acid, and which are at all times immersed in a water/ice bath. HCl is quantitatively solubilized in the acidic solution and forms chloride ions. The chloride ions are measured using ion chromatography. A rotameter downstream of the bubblers controls the gas flow to around 1-L/minute. A dry test meter downstream of the rotameter measures the absolute gas volume. The method used is similar to EPA Method 26 (40CFR Part 60 promulgated test method).

Samples were collected using four spargers connected in series. The first sparger was empty, the second and third each contained 15-ml of the dilute sulfuric acid solution. The fourth sparger contained a fresh charge of indicating silica gel, 6-16 mesh. Later samples collected at the G-19 location used both sulfuric acid and plain deionized water. The use of deionized water was investigated in order to reduce carbonate ion interference and to provide the lowest possible detection limit. A schematic of the sampling system is shown below in Figure 30.

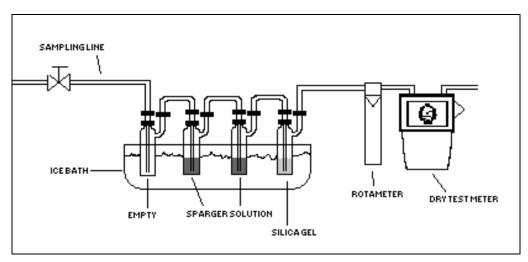


Figure 30 - Hydrogen Chloride (HCl) Sampling System

Possible interferents include other gaseous chlorine-containing species that could hydrolyze to chloride ions, such as  $ClO_2$  or  $NH_4Cl$ , or particulate matter containing chlorine. High levels of carbonate ion, likely to be present in syngas, can potentially interfere with the ion chromatography analysis. Careful selection of eluent pH and column type, depending on sample location, was necessary to reduce its impact.

Type 3 deionized water was used for all solution preparation, dilutions, and washing. The gas washing bottles (spargers) were soaked in dilute nitric acid and rinsed several times using deionized water prior to use. The sulfuric acid solution was prepared from ACS grade concentrated sulfuric acid (2.8-ml diluted to 1-liter with deionized water). Chloride standards were prepared from 1000-mg/L chloride IC standards purchased from Spex Corporation. 100-ml Class A volumetric flasks and an Eppendorf variable volumetric pipet was used to prepare standards and samples. Very dilute standards were prepared by serial dilution of higher concentrations. An independent QC check was run to verify instrument response using a standard purchased from Dionex Corporation. All standard concentrations are certified and NIST-

traceable. Unused samples were stored in 125-ml clear pre-cleaned I-Chem borosilicate glass bottles with Teflon sealed tops.

Samples were analyzed on a Dionex ICS-1000 isocratic ion chromatography system. A standard suppressed conductivity detector with a Dionex ASRS-Ultra background suppressor measured chloride responses. Dionex Chromeleon chromatography software was used for data acquisition, peak integration, and reporting. At least four levels of calibration were used for each analysis. The actual calibration concentrations depended on the expected analytical concentration in each sample. Reagent blanks were analyzed and while no chloride was found, an interfering peak was present in the initial Novel Gas Cleaning Optional Program test campaign (NGC-OPT1) runs. The interferent was identified as HSO<sub>4</sub>-, present due to the pH of the eluent. The analytical method was changed to eliminate the interference for the critical NGC-OPT2 test campaign runs wherein a much lower concentration of HCl was expected. As the information in Table 10 shows, the specific column and eluent used in the analysis changed in order to continually improve the method as different sample types were analyzed.

Test Campaign	Date	Column	Eluent	Flow Rate, ml/min
NGC-OPT1	3/8/05-3/9/05	AS14	8mM/1mM carb/bicarb	1.0
NGC-OPT2A	3/24/05-3/29/05	AS10	40mM NaOH	1.2
NGC-OPT2B	3/30/05-3/31/05	AS17	2mM NaOH	1.0

**Table 10 - Analytical Method Parameters for Chloride Analysis** 

The sampling methodology was validated by purging sampling points with dry nitrogen and reference gases containing a certified concentration of HCl (29.88 ppm HCl in N<sub>2</sub>, 2.95 ppm HCl in N<sub>2</sub>, and 0.578 ppm HCl in N<sub>2</sub>). Validation data are provided in Table 11. The validation blanks for the NGC-OPT2A test were taken at the end of the test campaign and it is believed that the sample lines were not given sufficient time to purge out the remaining HCl. Because of this, additional validation blanks for the NGC-OPT2B test that followed were taken at the beginning of the test campaign. These blanks are more representative of true field blanks. Validation standards were not taken at the G-19 sampling point because it was feared that the high concentration of HCl in the reference gas would remain in the lines and slowly permeate into the test syngas, resulting in biased data. The satisfactory results at the G-13 location proved the inertness of the Silcosteel®-CR coated sampling lines. It should also be pointed out that replicate impinger measurements were made during the first test campaign (NGC-OPT1) using spargers run side-by-side. The reproducibility of the impinger sampling with ion chromatography chloride measurements was demonstrated. Duplicate analyses at G-13 resulted in concentrations of 10.9 and 10.8 ppmv HCl.

The following approach was developed to estimate the background HCl concentration at any point during the test. Before testing commenced, when the system was being purged with nitrogen, the average HCl concentration present in a "clean" sample line was defined as the sum of the average of the 'true' field blanks measured before HCl exposure in the NGC-OPT2B test (i.e.,  $\sim 0.020$  ppmv or 20 ppbv). Likewise, after testing concluded, when the system was being purged with nitrogen, the average HCl concentration present in the sample lines was defined as the sum of the average of the 'true' field blanks measured after HCl exposure in the NGC-OPT2B test (i.e., 57.3 ppbv). Then, because sample flow through these lines was maintained throughout testing, it is reasonable to assume that a 37.3 ppbv of HCl "burden" accumulated in a linear fashion and that during testing, the background concentration of HCl at any time t, C(t), can be determined from the following equation:

### $C(t) = 20 + [37.3/T] \cdot t$

where T is the total time of syngas exposure (in hours) and t is the elapsed time, also in hours, from the start of testing to the time of interest.

Date	Validation	Validation	Certified	Analytical	Comment
	Point	Туре	Result	Result, avg	
NGC-OPT2A					
3/25/05	G-19	Blank		40 ppbv	Post-test blank
3/25/05	G-14	Blank		56 ppbv	Post-test blank
3/25/05	G-19	Blank		76 ppbv	Post-test blank (N=2)
NGC-OPT2B					
3/29/05	G-14	Blank		28 ppbv	Pre-test blank
3/29/05	G-19	Blank		< 20 ppbv	Pre-test blank
3/29/05	G-19	Blank		23 ppbv	Pre-test blank
3/29/05	G-19	Blank		< 20 ppbv	Pre-test blank

 Table 11 – Chloride Analysis Validation Data for the First and Second Test Campaigns

### 8.2 Hydrogen Sulfide and Carbonyl Sulfide Analysis

Gas samples for laboratory analysis of  $H_2S$  and COS were collected at G-8, G-13, G-14, and G-19 to provide backup and confirmatory data for the on-line Varian PFPD GC measurements. Each Sulfinert<sup>®</sup> coated sampling canister used was cleaned by washing with high purity (chromatography grade) n-hexane and acetone, followed by baking at 70°C under vacuum with periodic air purges for at least 12 hours.

Gaseous H<sub>2</sub>S and COS were measured in GTI's in-house analytical laboratory using a GC coupled with either a flame photometric detector (GC-FPD) or a pulsed flame photometric detector (PFPD), following ASTM D6228 and GTI SOP PP-108. This analysis determines sulfur species in gas samples at concentrations greater than 0.00005% by volume (0.05 ppmv or 50 ppbv). The GC-FPD system is an HP 5890 with a standard flame photometric detector. The GC-PFPD system is the S-Pro from OI Corporation (OIC), utilizing an Agilent 6890 GC with an OIC pulsed flame photometric detector. The S-Pro system also contains a fully integrated permeation system that continuously supplies quantitative gas standards for calibration and performance checks. The entire sample pathway of both GCs is Sulfinert<sup>®</sup> coated. The GC-FPD system uses a 60-meter, 0.53-mm i.d. Supelco SPB-1 column with a 1-meter pre-column. The GC-PFPD system uses a 60-meter, 0.32-mm i.d. Agilent/J&W DB-1 column.

Interferences are any co-eluting species that may quench the response of the sulfur signal. These are mainly the light gases such as methane, carbon monoxide, nitrogen, and carbon dioxide. These compounds must be adequately separated from the components of interest. The use of the PFPD system eliminates many of these common interferences by using emission time domain processing as well as wavelength filtering to improve sensitivity and selectivity. In the PFPD system photometric emissions from selected elements (e.g. carbon and sulfur) are characterized by different decay rates. By setting time gates, inter-element interferences are reduced or eliminated.

Each GC is conditioned daily by injections of a sulfur standard to passivate the system. Following this, a minimum of three (GC-FPD) or two (GC-PFPD) calibration checks are made at varying pressures for each component. The recovery for the consecutive calibration checks must be within  $\pm 10\%$ . Final sulfur component quantitation is done by external calibration consisting of a minimum of four calibration points comprising a second order calibration curve for each component. Each calibration curve must have an  $r^2 > 0.995$ . The calibration check runs are then added to the calibration curve, and the curve is checked to ensure it maintains an  $r^2 > 0.995$ . All standard concentrations are certified and NIST-traceable. The actual injected pressure (in torr or mm Hg) is used to correct all sample and standard data to an equivalent pressure basis. Each injection loop and/or range setting has its own calibration curve to avoid any variances in loop size or inertness. The sample loop size for G-8, G-13/G-14, and G-19 samples was 15- $\mu$ L, 100- $\mu$ L, and 1-mL, respectively.

GTI's SOP requires that the GC column maintain adequate separation of components. Adjacent components analyzed at similar concentrations must maintain a valley point less than 5% of the height of the smaller peak. If the peak area for a given compound exceeds the highest calibration area by more than 10%, the sample will be reanalyzed either at a lower injection pressure, smaller injection volume, or adjusted detector range to obtain a peak area within the calibration range. The actual pressure that was injected is recorded.

The sampling methodology was validated during the NGC-OPT2 test campaign by purging sample points with dry nitrogen and reference gases containing a certified concentration of  $H_2S$ . Satisfactory results were obtained at G-13 and G-14, which proved the inertness of the Silcosteel<sup>®</sup>-CR coated sampling lines. Validation standards were not taken at G-19 because it was feared that the high concentration of  $H_2S$  in the reference gas would remain in the lines and slowly permeate into the test gas, resulting in biased data. Validation blanks taken at G-14 and G-19 consistently measured below the analytical method detection limit of 50 ppbv.

## 9. TEST MATERIALS

Pilot-scale testing materials stocks were determined based on estimates of Novel Gas Cleaning Process flows, test campaign durations, and other considerations such as cost and availability. Test materials included a relatively low-sulfur carbonaceous feedstock (metallurgical coke, Indian coal, and lignite), trona (and nahcolite as an alternative material) for bulk HCl removal in the Conditioning Filter-Reactor, BASF's R5-12 catalyst or Süd -Chemie's G-72E catalyst as a ZnO sulfur guard bed material, T-46 PROX-SVERS inert balls (92-95% alumina; 9.5, 19, and 38.1 mm (3/8, <sup>3</sup>/<sub>4</sub>, and 1-1/2 inch) in diameter) from Christy Companies in St. Louis, MO as support balls for the ZnO catalyst in the Sulfur Guard Bed, Süd -Chemie's G-72E and G-92C catalysts as Stage II desulfurization and dechlorination sorbents, respectively. These latter two materials are available commercially as 4.8 mm (3/16 in) extrudates and would require grinding to -325 mesh for injection into the syngas upstream of the Test Filter-Reactor. A suitable pulverizer was acquired for this purpose (LC-140 Fritsch Rotor-Speed Mill from the Gilson Company with an automatic vibrating feeder and a bag collection kit). The pulverizer made it possible for sorbent sizing to take place immediately before use, thus making sure sorbent properties were not adversely affected.

#### 9.1 Carbonaceous Feedstocks

The proximate analysis, ultimate analysis, heating value, and size distribution properties are summarized in Table 12, Table 13, and Table 14, for the metallurgical coke (Bethlehem Coke Breeze), Indian coal, and lignite coal, respectively. These analyses were performed on equal-weight composite samples from several supersacks. Bethlehem Coke Breeze is typically used as a startup material for the Flex-Fuel gasifier, but was included in this PDU test program given its relatively high chloride content which made it a preferred candidate for the first test campaign focusing on HCl removal in the Conditioning Filter-Reactor. The washed Indian coal was available from a recent project completed at GTI.<sup>13</sup>

The lignite coal was procured specifically for this program. Saskatchewan lignite (Shand) was first gasified during the latter stages of the second test campaign, where its suitability was confirmed as the feedstock of choice for the third test campaign. The third test campaign, which focused on the evaluation of mercury capture with sorbents at relatively high temperatures, used lignite exclusively as the source of syngas and provided another opportunity to demonstrate efficient gasification of lignite in the fluidized bed gasifier. The properties (proximate analysis, ultimate analysis, mineral ash analysis, etc.) of the processed coal are shown in Table 14 and in Table 15 for the raw coal (i.e., prior to drying and sizing, as provided by Great River Energy).

Arrangements were made with the Green River Energy Coal Creek Station (GRE/CSS) to have about 100 tons of Saskatchewan lignite processed (crushed, screened, and dried) to meet the Flex-Fuel gasifier feed specifications. GRE's fluidized-bed drying process produced three streams: product, elutriates, and undercuts, with a surface moisture of ~ 0% and inherent moisture of ~ 20%. To preserve the quality of the lignite and more importantly the contaminant levels for the NGC PDU test program (especially mercury content), the product and elutriates fractions were blended and the undercuts recrushed, screened, and stored in proper containers for additional blending, if necessary.

All three carbonaceous feedstocks have relatively low sulfur contents (0.56 to 0.89 wt.% S). Both the washed Indian coal and the North Dakota Lignite are suitable feedstocks for the second and third test campaigns. Each of these feedstocks contains very low chlorine so that trona injection in Stage I of the NGC Process could be foregone in these campaigns and focus could be directed primarily at the contaminant removal in Stage II of the process. Also, each feedstock contains sufficient mercury for the third test campaign.

#### Table 12 - Properties (Proximate Analysis, Ultimate Analysis, Screen Analysis, Heating Value, and Bulk Density) of Metallurgical Coke (Bethlehem Coke Breeze)

Proximate Analysis		(As Received)			
	Moisture, %	0.40			
	Volatile Matter, %	2.71			
	Ash (950C), %	13.57			
	Fixed Carbon, % (by difference)	83.32			
Ultimate Analysis		(Dry Basis)			
	Ash (950C), %	13.56			
	Carbon, %	82.42			
	Hydrogen, %	0.20			
	Nitrogen, %	1.11			
	Sulfur, %	0.69			
	Mercury, micro g/g	0.02			
	Chlorine, micro g/g	800			
	Oxygen, %	1.98			
	(by difference)				
Heating Value		(Dry Basis)	Fusion Temperature of	Ash (ASTM D1857)	, deg.F
				Reducing	Oxidizir
	BTU/lb	12,320			
			Initial Deformation (I'	2220	2,46
			Softening (ST)	2325	2,50
			Hemispherical (HT)	2340	2,54
	1		Fluid (FT)	2500	2,60

rissays by 505 Millerais	Services (through GTI CRS)					
Proximate Analysis		(As Received)		(As Received)	(Dry basis	
				w/SO3 correction w/	SO3 correction	
	Moisture, %	9.97		9.97		
	Volatile Matter, %	26.04		26.04	28.89	
	Ash (750C), %	31.64		31.56	35.02	
	Fixed Carbon, %	32.45		32.53	36.09	
	(by difference)					
Ultimate Analysis		(Dry Basis)				
	Ash (750C), %	35.02				
	Carbon, %	48.48				
	Hydrogen, %	3.09				
	Nitrogen, %	1.01				
	Sulfur, %	0.56				
	Mercury, micro g/g	0.14				
	Chlorine, micro g/g	< 100				
	Oxygen, %	11.84				
	(by difference)					
Heating Value		(Dry Basis)		Fusion Temperature of	Ash (ASTM D1857), F	
				-	Reducing	Oxidi
	BTU/lb	8,310				
				Initial Deformation (I'	> 2,700	> 2
				Softening (ST)	> 2,700	> 2
				Hemispherical (HT)	> 2,700	> 2
				Fluid (FT)	> 2,700	> 2
Screen Analysis	Reta	ained on Wt.	%			
		6	5.0%			
		12	5.0% 13.1%			
		20	13.1% 18.1%			
		20 40	18.1% 20.2%			
		40 60	15.0%			
		80	9.3%			
		100	9.5% 4.9%			
		100	4.9% 6.9%			
		200 230	3.9% 1.1%			
		230 270	1.1% 0.8%			
		325	0.8%			
		325 PAN	0.5% 1.2%			
	Tota	al	100.0%			
Bulk Density	1 01	9 g/ml				
Dum Density		5 lb/cu ft				

# Table 13 - Properties (Proximate Analysis, Ultimate Analysis, Screen Analysis, Heating Value, and Bulk Density) of Washed Indian Coal

Assays by GTI Chemical	Research Services Departmen	nt				
Proximate Analysis		(As Receiv	ed)	(As Received) w/SO3 correction w/s	(Dry Basis) SO3 correction	
	Moisture, %	18.62		18.62		
	Volatile Matter, %	35.49		35.49	43.61	
	Ash (750C), %	11.54		10.27	12.62	
	Fixed Carbon, %	34.35		35.62	43.77	
	(by difference)					
Ultimate Analysis		(Dry Basis)	)			
	Ash (750C), %	12.62				
	Carbon, %	60.38				
	Hydrogen, %	3.93				
	Nitrogen, %	0.92				
	Sulfur, %	0.89				
	Mercury, micro g/g	0.06		(0.000006) pct		
	Chlorine, micro g/g	122		(0.0122) pet		
	Oxygen, %	21.25		(0.0122) per		
	(by difference)	21.20				
Heating Value		(Dry Basis)	)	Fusion Temperature of	Ash (ASTM D1857),	F
-				-	Reducing	Oxidi
	BTU/lb	10,200				
				Initial Deformation (I'	2,065	2
				Softening (ST)	2,080	2
				Hemispherical (HT)	2,105	2
				Fluid (FT)	2,120	2
Screen Analysis	Ret	ained on	Wt. %			
		6	9.0%			
		12	23.8%			
		20	26.3%			
		40	20.5%			
		60	11.6%			
		80	4.5%			
		100	1.3%			
		140	1.0%			
		200	0.4%			
		230	0.4%			
		270	0.1%			
		325	0.1%			
		PAN	0.1%			
		Total	100.0%			
Bulk Density	0.83	37 g/ml				
	0.85	3 lb/cu ft				

## Table 14 - Properties (Proximate Analysis, Ultimate Analysis, Screen Analysis, Heating Value, and Bulk Density) of Processed North Dakota Lignite

	Saskatchewan Lignites						
	Cuchatorionan Eignicoo		Boundary Dam	Poplar River	Shand		
	Proximate Analysis As Rec.		200				
	Moisture, %		35.00	37.78	33.54		
	Ash, %		9.47				
	Volatile, %		24.82				
	Fixed Carbon, %		30.71				1 j/g = 0
	HHV, BTU/#		6,728				75
	HHV, j/g		15,676	,			
	Sulfur, %		0.54	,			1 btu/lb =
	MAF BTU/#		11,433				
	MAF j/g		26,639				
	Mercury, ppb		79				
	Chlorine, ppm		10.2	. 11.4	7.9		
	Ultimate Analysis As Rec.						
	Moisture, %		35.00	37.78	33.54		
	Carbon, %		41.70				
	Hydrogen, %		2.61				
	Nitrogen, %		0.79				
	Sulfur, %		0.54				
	Ash, %		9.47				
	Oxygen, %		9.90	9.87	9.70		
						Boundary Dam Poplar River	Shand
	Mineral Ash Analysis Dry	Factor				Elemental Concentrations. Ppm in coal	
SiO2	Silicon Dioxide, %	2.14	34.57			15299.38 22770.	
AI2O3	Aluminum Oxide, %	1.89	15.25			7640.91 15096.	
ΓiO2	Titanium Dioxide, %	1.67	0.50			282.75 493.	
FeO	Iron Oxide, %	1.29	5.02			3693.79 5562.	
CaO	Calcium Oxide, %	1.40	15.53			10508.55 13705.	
MgO	Magnesium Oxide, %	1.66	3.66			2092.03 4739.	
<b>&lt;</b> 20	Potassium Oxide, %	1.42	0.87			580.61 846.	
Va2O	Sodium Oxide, %	1.35	3.70			2596.26 292.	
SO3	Sulfur Trioxide, %	2.50	15.19			5752.50 50	
P2O5	Phosphorus Pentoxide, %	2.29	0.49			203.94 25.	
SrO	Strontium Oxide, %	1.18	0.30			242.94 143.	
BaO	Barium Oxide, %	1.12	0.23			199.23 430.	
	Undetermined, %		4.68	3.12	1.09	50907.13 30892.	53

 Table 15 - Proximate, Ultimate, and Mineral Ash Analyses for Saskatchewan Lignites (Provided by Great River Energy)

#### 9.2 Sulfur and Halide Sorbent Materials

Table 16 provides the chemical analyses, measured BET N<sub>2</sub> surface area, and the theoretical sulfur or chloride capacity for the PDU test program sulfur and halide sorbents. For reference, the laboratory counterparts for the Stage I bulk HCl removal sorbents are also included. Details on the NGC Process sulfur and halide sorbents were provided in the Base Program final report.<sup>1</sup> For the laboratory work, the Stage I bulk HCl removal sorbents were obtained from FMC in Green River, Wyoming (trona) and from the White River Nahcolite Minerals (WRNM) in Meeker, Colorado (nahcolite). For the PDU test program, Solvay Minerals in Houston, Texas was selected as the commercial supplier for both materials. Chemical analyses in Table 16 indicate both Solvay trona and nahcolite materials contain slightly less sodium than their laboratory counterparts, leading to lower theoretical chloride capacities. According to the information provided by the supplier, the Solvay T-200® contained 97.5% Na<sub>2</sub>CO<sub>3</sub>•NaHCO<sub>3</sub>•2H<sub>2</sub>O (i.e., 97.5% purity), 0.01% free moisture, 2.3% H<sub>2</sub>O insoluble material, and 0.1% NaCl. The bulk density is 49.0 lbs/ft<sup>3</sup>. The particle size distribution is as follows: 75% by weight < $70 \,\mu\text{m}$ ,  $50\% < 50 \,\mu\text{m}$ , and  $10\% < 6 \,\mu\text{m}$ . The Solvay sodium bicarbonate (nahcolite) contained > 99.9% NaHCO<sub>3</sub>. Its (loose) bulk density is 71.7 lbs/ft<sup>3</sup>. The particle size distribution is as follows: 82.1% by weight > 45  $\mu$ m (+325 mesh), 26.8% > 75  $\mu$ m (+200 mesh), and 0.5% > 150  $\mu$ m (+100 mesh). Both commercial trona and nahcolite materials were deemed to have suitable size distributions for the PDU test program and no additional grinding or sieving was performed on the Solvay bulk HCl removal sorbents.

Appropriate quantities of the G-92C (Stage II dechlorination sorbent) and G-72E (Stage II desulfurization sorbent) were procured from Süd-Chemie as extrudates. Sufficient amounts of each material were finely ground using the Gilson LC-140 Fritsch Rotor-Speed Mill. Preparation of the Stage II materials for injection into the conditioned syngas stream was performed in two steps. In a first step, each batch was ground to pass a 100 mesh screen, and in a second step this fine material was ground further to pass a 325 mesh screen. Finely-ground materials were stored in proper containers until use.

		Chemical Analysis (wt. %)					Theoretical Capacity (g Cl or S/100 g)	BET N <sub>2</sub> Surface Area (m <sup>2</sup> /g)
	Na	Zn	С	Н	Al	Ca		
Trona								
(Na <sub>2</sub> CO <sub>3</sub> •NaHCO <sub>3</sub> •2H <sub>2</sub> O								
Solvay T-200®	31.63		6.92	1.81			48.8	
FMC (Green River, Wy)	35.6		10.68	1.84			54.9	8.65
Nahcolite (NaHCO <sub>3</sub> )								
Solvay Sodium Bicarbonate	23.9		10.01	0.74			36.9	
WRNM (Meeker, CO)	26.9		14.07	1.38			41.5	9.69
<b>G-92C</b> (Na <sub>2</sub> O/Al <sub>2</sub> O <sub>3</sub> )	6.41				39.6		9.9	165
Süd-Chemie (Louisville, Ky)								
(two-stage grinding at GTI)								
G-72E (ZnO/calcium		69.8			4.18	1.17	34.2	40.3
aluminate)								
Süd-Chemie (Louisville, Ky)								
(two-stage grinding at GTI)								

Table 16 – Novel Gas Cleaning Process Sulfur and Halide Sorbents

#### 9.3 Mercury Sorbent Materials

Selection and laboratory evaluation of candidate mercury-removal sorbents was completed earlier in the project, identifying two candidate sorbents with potential to capture mercury to levels below 0.01 ppb in the NGC Process configuration. The first sorbent, intended as the lower temperature mercury removal sorbent, was acquired from Synetix/Johnson Matthey (JM) in a pre-sulfided form. This mixed metal oxide (CuO and ZnO) sorbent was designated as GTI-Hg-S9. Characterization work at GTI indicated the sorbent contained 32.4% Cu, 19.9% Zn, 14.3% S, and 8.56% Al, and had a BET nitrogen surface area of 71.1 m<sup>2</sup>/g. The second sorbent was provided by TDA, as an improved version of an earlier mercury sorbent that was evaluated in the laboratory portion of this program under a non-disclosure agreement. TDA's mercury removal sorbent (also consisting of CuO, ZnO, and Al<sub>2</sub>O<sub>3</sub> as the primary components in addition to some minor promoters) was provided as 150-250 µm powder and required some additional activation, as discussed below, prior to further grinding at GTI.

TDA provided a 10-kg batch of their mercury sorbent. Approximately 6.8 kg of this batch, in the form of weak pellets, was provided in a separate container. The remainder was provided separately in the form of stronger pellets. The chemical compositions of both materials are identical. TDA requested that we use the weak pellet batch first. In addition, TDA specified that their sorbent requires activated prior to use. According to the information provided, activation is typically carried out at 230°C using hydrogen as the reducing gas. Typically, 2% hydrogen in an inert carrier gas is used, with total usage of 20 grams of hydrogen per kilogram of sorbent. Flow rates of activation gas for small quantities are usually 100 sccm/g for eight hours. This equates to 800 sLpm/kg divided by the desired number of hours of flow. Following activation, the sorbent is kept under an inert or reducing atmosphere until use. Ideally, it is preferable first to grind the sorbent to the required size (i.e., < 325 mesh), and then to activate the resulting sorbent fines. In this work, however, given the sorbent quantities involved, it was necessary to activate the TDA sorbent while still in pellet form, and then to finely grind the resulting activated sorbent. Every effort was made to minimize sorbent exposure to the atmosphere during this process.

## 10. NOVEL GAS CLEANING OPTIONAL PROGRAM TEST CAMPAIGN #1 (NGC-OPT1)

#### 10.1 Objectives

The primary objective of the Novel Gas Cleaning Program Test Campaign 1 (NGC-OPT1) was to evaluate the performance of the Conditioning Filter-Reactor (CF-R, T-2153) for combined particulate/halide (mainly HCl) removal from syngas derived from the gasification of a carbonaceous feedstock. This was accomplished through a series of test segments that were designed to:

- set the Flex-Fuel gasifier operating conditions for producing syngas from the test feedstocks (Bethlehem Coke Breeze and washed Indian coal) at 200 to 225 psig pressure, preferably with a face velocity of at least 3 ft/min in the T-2153 CF-R; this corresponds to approximately 2,700 lbs/hr of syngas.
- determine the operating conditions for the Direct Spray Water Quench (DSQ) system, the CF-R (including pulsing), and the sorbent (trona) injection system to achieve 1 to 5 ppmv of HCl and ~ < 0.1 ppmw particulate content in the T-2153 CF-R product syngas, at an operating temperature of at least 830°F (preferably).</li>
- 3. confirm the effectiveness of "conventional" syngas Sample Extraction & Conditioning (SE&C) systems at sample points G-8 (CF-R inlet) and G-13 (CF-R outlet), using two carbonaceous feedstocks with different chlorine contents (~ 800 ppmw for Bethlehem Coke Breeze and ~ 135 ppmw for washed Indian Coal), with and without sorbent injection for HCl capture.

4. provide "cold" and "hot/warm" slipstream samples from G-8 and G-13 for comprehensive, continuous, sensitive, and accurate measurements by personnel from the GTI Birmingham Office, as part of their project scope to develop an analytical suite as a key enabling technology for gasification systems integration.

### 10.2 Process Flow Diagram for NGC-OPT1

For this test series in the Flex-Fuel Test Facility (FFTF), the syngas flow in the NGC Conditioning Section was as shown in Figure 31.

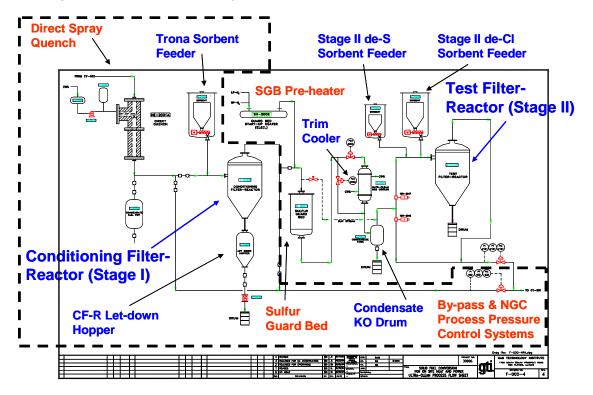


Figure 31 – Flow Diagram of NGC Process Section in the Flex-Fuel Test Facility (area delineated by dashed lines indicates equipment used in NGC-OPT1)

Raw syngas from the Secondary Cyclone (CY-401) was partially cooled in the DSQ system (HE-2001A) and was then directed to the T-2153 CF-R vessel. After passing through the filter elements or candles in the CF-R vessel, the syngas was routed through the Pressure Let-Down station (PIC-602), then to the Tertiary Cyclone (CY-601), and finally to the Flare (FL-601). As indicated above, the Conditioning Section also comprises a Sulfur Guard Bed (SGB) and a second Indirect Syngas Cooler (Trim Cooler) for bulk sulfur removal and temperature reduction of the CF-R product gas to meet the requirements of the inlet syngas to the Test Section. It was acceptable to by-pass the SGB in this test since the relatively low sulfur contents of the test feedstocks (0.69% for Bethlehem Coke Breeze and 0.55% for washed Indian coal) allowed operation while still meeting the EPA sulfur emissions requirements.

After pulsing cycles were established with the nitrogen blow-back system on the T-2153 CF-R vessel, HCl removal sorbent, trona (or nahcolite as an alternative sorbent, if necessary), was injected into the syngas stream, immediately before entering the T-2153 CF-R, through the T-2101 Sorbent Injection system. The latter combines a loss-in-weight solids feeder with dilute-phase flow in nitrogen to convey the sorbent into the syngas.

Representative samples of the syngas were extracted, conditioned (de-dusted, cooled, and depressured), and analyzed with suitable instruments at two main locations:

- G-8: Partially-cooled raw syngas (between DSQ and CF-R)
- G-13: Partially-conditioned T-2153 CF-R product syngas (essentially dust-free, dechlorinated to 1-5 ppmv HCl, but still containing all the raw syngas sulfur).

Other analytical work was also performed on various samples to estimate CF-R particulate leakage (at S-13C), to characterize gasifier performance and efficiency in generating syngas from the test feedstock, to characterize process emissions for permitting activities, and to assess disposal options for the gasification and syngas cleanup byproduct materials. Process samples included fuel feedstock, ash, dust, raw syngas, fresh sorbent materials, spent sorbents, and condensed liquids, in addition to several gas samples throughout the NGC Process section. Additional details are provided in the following sections.

### 10.3 NGC-OPT1 Sequence / Chronology

During the NGC-OPT1 campaign, the following sequence was followed to accomplish test objectives:

- 1. <u>System Heat up with Start-Up Heater (SH-201)</u>: Direct flue gas from Gasifier through DSQ, through T-2153 CF-R vessel, to pressure control valve(s), PCV-602.
- 2. Initiate solids feeding with metallurgical coke (Bethlehem Coke Breeze), for second-stage gasifier heating, for establishing a fluidized-bed of solids within the gasifier, and ultimately for establishing steady-state (SS) gasifier operation at 200-225 psig pressure.
  - 2.1. Adjust Gasifier operating parameters to achieve target gasifier pressure and target syngas flowrate (and face velocity for filter candles).
  - 2.2. Establish CF-R operating temperature at ~ 830°F or higher.
    - 2.2.1. Adjust DSQ system set point to achieve target T-2153 syngas outlet temperature
    - 2.2.2. TIC-2022 set point likely between  $\sim 900$  and 950°F
    - 2.2.3. TIC-2022 may be limited by thermal expansion of process piping
  - 2.3. Verify SE&C skid viability at the G-8 and G-13 sampling locations via spike-and-recovery methods (HCl ~ 7.7 ppmv; H<sub>2</sub>S ~ 535 ppmv (and COS ~ 65 ppmv)). Impinger samples and FT-IR (FT-IR has a nominal detection limit for HCl in the range of ~ 1 to 5 ppmv)
- 3. When a reasonable rate of syngas generation from the Gasifier has been established, continue operating at SS conditions to verify/establish the following:
  - 3.1. Measure HCl (and H<sub>2</sub>S & COS) levels in the raw syngas (at G-8) and establish variability
    - 3.1.1. Impinger samples and FT-IR at G-8 with syngas. (Important to establish range of variation in HCl using FT-IR in order to evaluate removal effectiveness with this instrument at G-13 (see below).)
    - 3.1.2. With G-8 hot-gas sample line to FT-IR inlet, monitor HCl concentration during pulsing on T-2153 to determine if nitrogen pulse affects measurement.
    - 3.1.3. Monitor raw gas composition at G-8 with mass spectrometer and GC/FPD/TCD. Determine spike duration requirements and recoveries for GC system (with H<sub>2</sub>S and COS spike).
  - 3.2. Establish preferred conditions for CF-R pulsing frequency in the 1-10 range to handle dust loading from the metallurgical coke.

- 3.2.1. Observe pressure-drop increase on the T-2153 CF-R and adjust pulsing as needed to give consistent "clean" differential pressure for filter candles.
- 3.2.2. Fine-tune Pulsing on the T-2153 CF-R to achieve cleaning at minimal nitrogen use
- 3.2.3. Pulsing of the T-2153 filter with the fast-acting valve
- 3.2.4. Pressure of Blow-Back system nitrogen in T-2304 Accumulator: starting point ~ twice process pressure (i.e., 400 to 450 psig)
- 3.2.5. Length of pulsing time with fast-acting valve: 0.2 to 0.3 sec
- 3.3. Measure HCl levels with impingers and FT-IR (and H<sub>2</sub>S & COS) in the raw syngas and CF-R product syngas (at G-8 and G-13) and establish that, with no trona sorbent being injected, inlet and outlet syngas halide (and sulfur) contaminant concentrations are essentially the same.
- 4. When preferred test conditions are satisfied as described under 3 above:
  - 4.1. Prepare to start trona injection into the syngas stream upstream of the CF-R vessel
    - 4.1.1. Set Transport Nitrogen rate (as controlled by differential pressure) required for adequate conveying of sorbent into process syngas.
    - 4.1.2. Start with ~ 50 ft/sec injection velocity (22 scfm nitrogen rate), yielding approximately 5 psi differential pressure.
    - 4.1.3. (Might study minimum pressure differential required for protecting sorbent-weighing system from gasifier cycles / upsets)
  - 4.2. Initiate bulk HCl removal sorbent (trona) injection at the maximum rate of Na/Cl=10 based on the syngas HCl content at G-8

Trona (Solvay T-200®): Chemical formula: Na<sub>2</sub>CO<sub>3</sub>•NaHCO<sub>3</sub>•2H<sub>2</sub>O Purity: 97.5% Bulk density: 49.0 lbs/ft<sup>3</sup> Size distribution: 75% < 70  $\mu$ m, 50% < 28  $\mu$ m, 10% < 6  $\mu$ m

Trona Injection Rate (grams/min) =  $[(453.6)*(226)/[(3)*(60)*(10^{6})]] * [[HCl]_o*Q_m/MW_{sg}] * R$ 

- [HCl]<sub>o</sub> = Measured HCl concentration in raw syngas (at G-8), ppmv
- $Q_m = Raw$ , partially-cooled syngas, lbs/hr
- MW<sub>sg</sub> = Molecular weight of raw, partially cooled syngas, lbs/lb-mole
- R = Na to Cl molar ratio

Example: Raw syngas at 3000 lbs/hr with an average MW of 25 and containing about 75 ppmv HCl, the trona injection rate required for a sodium-to-chlorine (Na/Cl) ratio of 10 is about 51.3 g/min (52.6 g/min for 97.5% purity for the Solvay T-200® trona material).

- 4.2.1. While allowing the system to stabilize (i.e., a somewhat constant fly ash/trona mixture composition of the filter cake), continue to measure HCl with impingers (and H<sub>2</sub>S & COS) levels in the CF-R inlet and outlet syngas (at G-8 and G-13 sampling locations).
- 4.2.2. Monitor filtered syngas composition at G-13 with GC/FPD/TCD.
- 4.2.3. Attach G-13 hot-gas sample line to FT-IR inlet to monitor HCl concentrations. Monitor HCl concentration during pulsing on T-2153 to determine how pulsing affects HCl removal. (Re-plumb FT-IR to sample from G-8 inlet after this test (1-2 hrs)).
- 4.2.4. If the CF-R product syngas HCl content (i.e., at G-13) is < 5 ppmv, continue operating at Na/Cl=10 for six (6) hours, which should result in at least 4-6 consistent samples. Note: If

we do not achieve < 5 ppmv at G-13 with Na/Cl=10, then examine the HCl sorbent fines/total fines ratio and adjust (increase) sorbent injection rate to achieve 0.1 to 0.4.

- 4.2.5. Transition to a lower sorbent injection rate (i.e., Na/Cl=5) and measure effect of lower sorbent rate on HCl in syngas with impingers at G-8 and G-13 (3-4 hours or about 3 consistent samples).
- 4.2.6. Attach G-13 hot-gas sample line to FT-IR inlet to monitor HCl concentrations. Monitor HCl concentration during pulsing on T-2153 to determine how pulsing affects HCl removal. Leave FT-IR sampling from G-13.
- 4.2.7. Shut off HCl sorbent injection and Transport Nitrogen; allow system to stabilize (pulse clean manually to accelerate sorbent removal) and measure HCl in syngas with impingers at G-8 and G-13 ( 3 hours or 3 consistent samples)
- 4.2.8. Measure (estimate) particulate leakage (at S-13)
- 4.3. Transition from Bethlehem Coke Breeze to washed Indian coal feeding to Gasifier
  - 4.3.1. Re-plumb FT-IR to sample from G-8 inlet.
  - 4.3.2. Continue to operate gasifier at 200-225 psig; measure HCl and (H<sub>2</sub>S and COS) at inlet (G-8) and outlet (G-13), with no trona sorbent injection (see note below); (3-4 hours or about 3 consistent samples).
  - 4.3.3. Adjust Gasifier operating parameters to achieve new target gasifier pressure (300 psig
  - 4.3.4. Adjust Gasifier bed-ash to target level; achieve high-carbon conversion
  - 4.3.5. Adjust CF-R pulsing frequency as necessary
  - 4.3.6. Collect samples in the Gasification Section for material and energy balances
  - 4.3.7. Measure HCl and (H<sub>2</sub>S & COS) at inlet (G-8) and outlet (G-13). Note: with the HCl content of the raw syngas expected to be  $\sim$  10 ppmv, no trona injection should be performed. The measurements taken would serve as another check on the viability of our SE&C skids and measurement instruments.

GC system samples to be collected from both G-8 and G-13 hot-gas sample lines.

#### **10.4 Results and Discussion - NGC-OPT1**

#### **Gasifier and Conditioning Filter-Reactor Operations**

This first test campaign concentrated on the performance of the Conditioning Filter-Reactor filtration and HCl removal. The salient characteristics of the gasification section (Flex-Fuel gasifier and Direct Spray Quench system) and the Conditioning Filter-Reactor of the NGC Process section are summarized in Table 17 and Table 18. Gasification of Bethlehem Coke Breeze was conducted successfully for ~ 53 hours, from about 12:00 noon on March 8 through 17:00 on March 10, at which time the feed to the gasifier was switched to washed Indian coal. Testing with washed Indian coal extended for ~ 9 hours, until about 02:00 on March 11 when the NGC-OPT1 test campaign was terminated. This test campaign has been divided into four (4) test segments as described in Table 17. Test segments 2, 3, and 4 represent, respectively, steady-state testing periods during operation with metallurgical coke without trona injection, metallurgical coke with trona injection, and Indian coal without trona injection. The bulk HCl removal sorbent (trona) was injected through the T-2101 Sorbent Injection System from about 23:00 on March 9 until about 17:00 on March 10, i.e., third test segment (1-TS3). Trona was injected into the syngas immediately upstream of the T-2153 CF-R vessel. As a precaution, trona injection during this campaign was shut off during pulsing of the T-2153 filter vessel.

Overall	
Start	3/8/2005 at ~ 12:00
End	3/11/2005 at ~ 02:00
Duration, hr	62
Start-up and Establishing Steady State with	
Metallurgical Coke (1-TS1)	
Start	3/8/2005 at ~ 12:00
End	3/8/2005 at ~ 18:00
Duration, hr	~ 6
<b>Operating at Steady State with Met Coke (1-TS2)</b>	
Start	3/8/2005 at ~ 18:00
End	3/9/2005 at ~ 23:00
Duration, hr	~ 29
Trona Sorbent Injection (1-TS3)	
Start	3/9/2005 at ~ 23:00
End	3/10/2005 at ~ 17:00
Duration, hr	~ 18
Transition to Washed Indian Coal (1-TS4)	
Start	3/10/2005 at ~ 17:00
End	3/11/2005 at ~ 02:00
Duration, hr	~ 9
Terminate NGC-OPT1	3/11/2005 at ~ 02:00

Table 17 - Test Segments in the NGC-OPT1 Test Campaign (March 7-11, 2005)

Throughout testing, the syngas temperature at the outlet of the Direct Spray Quench system was maintained consistently above 800°F (Table 18). The process conditions for the Conditioning Filter-Reactor were relatively constant for  $\sim$  47 hours, from about 18:00 hours on March 8 through the end of the gasification of metallurgical coke at 17:00 on March 10. The solids loading in the syngas was relatively low, and the pulsing cycles were relatively long (once every 2 to 4 hours). As the gasifier feed was switched from metallurgical coke to washed Indian coal, the syngas flow rate increased considerably, as did the solids loading in the syngas. The frequency of pulsing had to be increased to approximately 7-8 pulses per hour.

 Table 18 - Gasification and NGC Process Sections Operating Conditions during the NGC-OPT1

 Test Campaign

	Met Coke	Indian coal (washed)
Start	3/8/2005 at ~ 18:00	3/10/2005 at ~ 17:00
End	3/10/2005 at ~ 17:00	3/11/2005 at ~ 02:00
Duration, hr	47	9
Gasifier		
Pressure, psig	205	210
Bed Temp., °F	1800 - 1875	1625 - 1725

Outlet Temp., °F	1675 - 1725	1600 - 1625
Bed Height, ft	8-11	6 - 9
Syngas Temp. at Secondary Cyclone, °F	1400 - 1450	1425 - 1450
Direct Spray Quench (DSQ)		
DSQ Inlet Temp., °F	1400 - 1450	1425 - 1450
DSQ Outlet Temp., °F	800 - 875	800
Quench Rate, lb/hr	0 - 150	275 - 325
Syngas Flowrate, lbs/hr	1800 - 2000	2300 - 2700
Pressure, psig	200	200
Conditioning Filter-Reactor (CF-R)		
CF-R Inlet Temp., °F	710 - 750	> 750
CF-R Outlet Temp., °F	670 - 690	690 - 710
Pressure, psig	200	200
Pulsing Frequency, times/hr	0.3 – 0.5	7 - 8

Temperature drop and pressure drop behaviors, and key process parameters (inlet syngas temperature, syngas mass flowrate, and operating face velocity) for the CF-R vessel are reported in Figure 32 and Figure 33 for the NGC-OPT1 campaign. The figures shown cover the entire test duration, and on each figure the various test segments are delineated consistent with the information provided in Table 17. The Flex-Fuel gasifier pressure was brought up to ~ 200 psig after initiating the gasification of Bethlehem Coke Breeze (during test segment 1-TS2). The inlet temperature for the syngas to the CF-R was maintained consistently above 800°F throughout testing. Process conditions were relatively constant for the CF-R vessel, during each of the three test segments shown on the figures. The syngas mass flowrate at the CF-R inlet was quite stable, averaging ~ 2,740 lbs/hr, which corresponds to approximately 2.18 ft/min operating face velocity. Both the syngas flowrate and face velocity increased during the final test segment of the NGC-OPT1 campaign, when the gasifier feed was switched from metallurgical coke to the more reactive washed Indian coal.

As shown in Figure 32, during metallurgical coke operation (i.e., test segment 1-TS2) the pulse cleaning frequency was low and the filter pressure drop behavior was relatively stable, with about 89 minutes between pulse cleaning events due to the relatively low gas flow rate and the low metallurgical coke ash content. The baseline pressure drop was 25-30 inches of water (in wg), and the peak pressure drop was about 50 in wg, with the pressure recovery on cleaning being about 20 in wg. The gas temperature drop experienced across the filter vessel was 175 to 250°F. This large temperature drop is due to the small vessel size and the low syngas flow rate through the vessel.

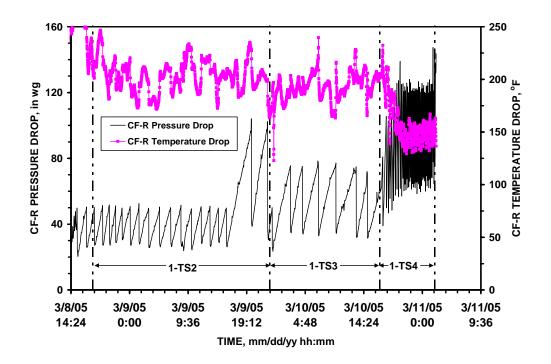


Figure 32 – Test Campaign #1 Conditioning Filter-Reactor Pressure and Temperature Drop

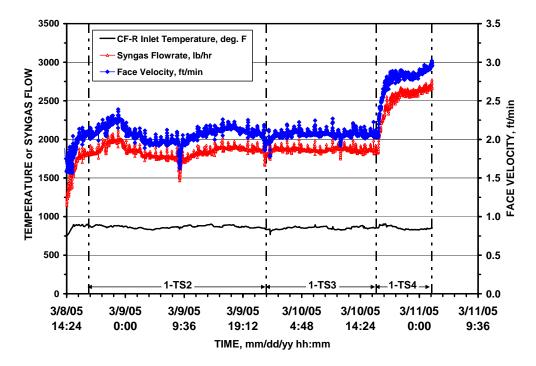


Figure 33 – Conditioning Filter-Reactor Inlet Syngas Temperature, Mass Flowrate, and Face Velocity during Test Campaign #1

When trona feeding was included in the operation (i.e., 1-TS3), the pulse cleaning frequency was reduced further to about 154 minutes between pulse cleanings (this is an operating parameter selection), resulting in a pressure drop recovery of about 40 in wg. The baseline pressure drop was about 36 in wg, and the peak pressure drop was about 71-77 in wg. The gas temperature drop experienced across the filter vessel was 173 to 225°F. It does not appear that the injected Trona resulted in much additional filter pressure drop.

During the final period of operation with washed Indian coal without Trona injection (i.e., 1-TS4), the pulse cleaning frequency was much higher and the filter pressure drop behavior was a little less stable, with about 10 minutes between pulse cleaning events due to the high Indian coal ash content. The baseline pressure drop was 47-83 in wg, increasing slowly with time. The peak pressure drop was about 106-159 in wg, with the pressure recovery on cleaning being about 50-60 in wg. The gas temperature drop experienced across the filter vessel was lower at 130 to 160°F due to the increased mass flow of Indian coal ash.

#### **Bulk HCl Removal**

The high-temperature process interface approach for syngas sample extraction and conditioning (illustrated in Figure 19 and in Figure 20) was validated at G-8 (CF-R inlet) and at G-13 (CF-R outlet) process locations. For the process conditions encountered, the sample gas was maintained at temperatures above 400°F, the pressure of the gas was reduced from 200 psig to 1-2 psig, and no dilution was required to prevent condensation into the FT-IR or mass spectrometer on-line instruments. A Nafion<sup>®</sup> dryer was used on the sample syngas stream ahead of the gas chromatographs to protect the columns from water in the sample gas.

Using a certified 7.7 ppmv HCl-N<sub>2</sub> gas mixture and spike-and-recovery techniques, EPA Method 26 impingers were used to collect samples from the high-temperature process interface sample lines from a tee at the inlet to the on-line instruments. Concentrations of HCl measured (using ion chromatography) 8.1, 7.7, and 6.8 ppmv HCl from these spike samples, yielding an average 100% recovery (see sample ID #'s 051119-038, -039, and -040 in Table 19 below, which provides a summary of all impinger/ion chromatography analyses made during the NGC-OPT1 test campaign). Reproducibility of the impinger sampling/ion chromatograph chloride measurements was demonstrated. Duplicate (simultaneous) impinger samples from the second SE&C Skid at the G-13 location measured 10.9 and 10.8 ppmv HCl (see sample ID #'s 051119-129 and -130 in Table 19).

The syngas HCl content at the Conditioning Filter-Reactor inlet, as measured at the G-8 sampling location, is displayed in Figure 34 as a function of time over the total test campaign duration. At first glance it appears that the inlet HCl content was quite variable over the test period using metallurgical coke, ranging from 10 to 105 ppmv. These variations, however, have been determined to be caused by char interferences of the first sample extraction and conditioning skid. As noted earlier, the SE&C Skid #1 comprises two gas trains each containing one Mott filter (A and B). During data collection from the beginning of the NGC-OPT1 until about 13:15 on 3/10/2005, Filter A was being used to remove char from the syngas slipstream. As char collected on the filter surface, the measured HCl concentration gradually decreased as, apparently, HCl was being removed through reaction (adsorption and/or chemisorption) with the char. A post-test sample from Filter A on the SE&C Skid #1 (at G-8) was analyzed for chloride and determined to contain about 0.014 % Cl (and 0.56% S).

To confirm the expected HCl concentration in the product raw syngas, the char-laden syngas slipstream at G-8 was directed through Filter B, the Mott filter housing of which had previously been thoroughly cleaned. As shown in Figure 34, immediately after switching, the measured HCl concentration increased from approximately 27 ppmv to 106 ppmv (see sample ID # 051119-132 in Table 19 taken within 30 minutes of filter switching). This value closely confirmed the expected HCl concentration in the product raw syngas, shown as a horizontal solid line in Figure 34. At a coke feed rate of approximately

372 lb/hr, a coke feed assaying about 800 μg Cl/g (i.e., 0.08 wt. % Cl or 800 ppmw Cl, Table 12), a product syngas mass flowrate of about 1,853.24 lb/hr, and an average MW of 24.6 for the product syngas, the HCl concentration in the wet syngas (after the DSQ system) was expected to contain about 111.3 ppmv HCl during the operating period from 18:00 on 3/8/2005 to 17:00 on 3/10/2005. Therefore, the inlet HCl concentration is considered to be about 111 ppmv for the purpose of estimating HCl removal efficiency in the CF-R. The washed Indian coal (~ 596 lb/hr coal feed to gasifier, ~ 2,509 lb/hr of syngas, and 100-140 ppmw Cl, Table 13) should generate about 16-23 ppmv HCl in the syngas, sufficiently low to forego trona injection upstream of the CF-R vessel.

Ref	Lab Sample ID	Sample Point	Date	Time Period	Average Time	Test	Measured HCI, ppmv
					(min)	Segment	
1	051119-022	G-8 Validation Test #1		-	-	•	1.7
2	051119-023	G-8 Validation Test #2		-	-		3.5
3	051119-038	G-13 Validation Test #1		-	-		8.1
4	051119-039	G-13 Validation Test #2		-	-		7.7
5	051119-040	G-13 Validation Test #3		-	-		6.8
						4 500	100
6 7	051119-045	G-13	3/9/2005	04:29 - 04:58	29 20	1-TS2	15.9
8	051119-046 051119-047	G-13 G-13	3/9/2005 3/9/2005	05:20 - 05:59 06:02 - 06:39	39 37	1-TS2 1-TS2	8.9 7.9
9	051119-047	G-15 G-8	3/9/2005	06:57 - 07:31	34	1-132 1-TS2	38.5
10	051119-055	G-8	3/9/2005	07:36 - 08:09	34	1-132 1-TS2	54.2
10	051119-055	G-8	3/9/2005	08:14 - 08:45	31	1-TS2 1-TS2	77.4
12	051119-058	G-8	3/9/2005	10:14 - 10:47	33	1-TS2	67.1
13	051119-064	G-13	3/9/2005	10:56 - 11:27	31	1-TS2	10.6
14	051119-065	G-13	3/9/2005	11:34 - 12:07	33	1-TS2	20.9
15	051119-066	G-13	3/9/2005	12:14 - 12:43	29	1-TS2	22.2
16	051119-069	G-13	3/9/2005	12:54 - 13:24	30	1-TS2	27.0
17	051119-071	G-13	3/9/2005	14:48 - 15:19	31	1-TS2	18.3
18	051119-072	G-13	3/9/2005	15:24 - 16:00	36	1-TS2	21.5
19	051119-073	G-8	3/9/2005	16:34 - 17:04	30	1-TS2	20.0
20	051119-081	G-8	3/9/2005	17:24 - 17:54	30	1-TS2	11.7
21	051119-089	G-8	3/9/2005	22:19 - 22:51	32	1-TS2	88.4
22	051119-090	G-8	3/9-10/05	23:45 - 00:20	35	1-TS3	35.4
23	051119-097	G-13	3/10/2005	00:54 - 01:34	40	1-TS3	12.4
24	051119-098	G-8	3/10/2005	01:40 - 02:12	32	1-TS3	30.4
25	051119-099	G-13	3/10/2005	02:20 - 02:43	23	1-TS3	45.8
26 27	051119-105	G-8 G-13	3/10/2005 3/10/2005	03:07 - 03:30	23 32	1-TS3 1-TS3	12.7 41.0
27 28	051119-106 051119-111	G-13 G-8	3/10/2005	03:38 - 04:10 04:15 - 04:46	32	1-155 1-TS3	41.0
28 29	051119-112	G-13	3/10/2005	05:15 - 05:41	26	1-133	33.9
30	051119-112	G-15 G-8	3/10/2005	05:48 - 06:17	20 29	1-155	41.3
31	051119-114	G-13	3/10/2005	06:22 - 06:45	23	1-TS3	14.5
32	051119-115	G-13	3/10/2005	07:03 - 07:27	24	1-TS3	19.6
33	051119-118	G-8	3/10/2005	07:34 - 07:59	25	1-TS3	38.3
34	051119-119	G-13	3/10/2005	08:04 - 08:30	26	1-TS3	17.4
35	051119-121	G-8	3/10/2005	08:50 - 09:28	38	1-TS3	13.2
36	051119-128	G-13	3/10/2005	10:32 - 11:05	33	1-TS3	18.5
37	051119-129	G-13 (A, DTM-1)	3/10/2005	11:26 - 11:56	30	1-TS3	10.9
38	051119-130	G-13 (B, DTM-2)	3/10/2005	11:20 - 11:56	36	1-TS3	10.8
39	051119-131	G-8	3/10/2005	12:33 - 13:00	27	1-TS3	20.4
40	051119-132	G-8	3/10/2005	13:44 - 14:13	29 20	1-TS3	106
41	051119-143	G-8	3/10/2005	16:17 - 16:47	30	1-TS3	69.9 18.7
42 43	051119-146	G-13 G-13	3/10/2005 3/10/2005	16:56 - 17:25	29 19	1-TS4 1-TS4	18.7 10.6
43	051119-147 051119-148	G-13 G-13	3/10/2005	17:37 - 17:56 18:02 - 18:17	15	1-154 1-TS4	10.6
44	051119-148	G-13 G-8	3/10/2005	19:38 - 20:08	15 30	1-154 1-TS4	11.8
45	051119-164	G-13	3/10/2005	20:15 - 20:47	30	1-154 1-TS4	13.6
40	051119-165	G-13	3/10/2005	21:50 - 22:13	23	1-154 1-TS4	9.4
48	051119-166	G-15 G-8	3/10/2005	23:02 - 23:27	25	1-154 1-TS4	9.2
49	051119-178	G-13	3/11/2005	00:00 - 00:27	27	1-TS4	7.5
50	051119-179	G-8	3/11/2005	01:00 - 01:25	25	1-TS4	5.1
51	051119-180	G-13	3/11/2005	02:01 - 02:22	21	1-TS4 1-TS4	9.8
52	051119-190	Impinger Field Blank	3/11/2005	10:30		1-1.54	< 1.0

Table 19 - Summary of Impinger/Ion Chromatography Analyses during the NGC-OPT1 Test
Campaign

The EPA Method 26 impinger/ion chromatograph batch HCl sampling and measurements and online FT-IR HCl analyses showed very good agreement. Both approaches were able to identify and quantify the effect of char on HCl concentrations in the syngas. The FT-IR measurements, superimposed on the batch measurements in Figure 35, also showed a marked drop in HCl levels and clearly indicated char was a significant factor in this reduction. The data indicated a very rapid and significant response to char. As shown in Figure 35, immediately on switching from Filter A to Filter B, the on-line FT-IR measured HCl > 100 ppmv. The peak value of HCl measured was consistent with the maximum HCl concentration expected in the process gas based on an analysis of chloride content in the fuel, as discussed above.

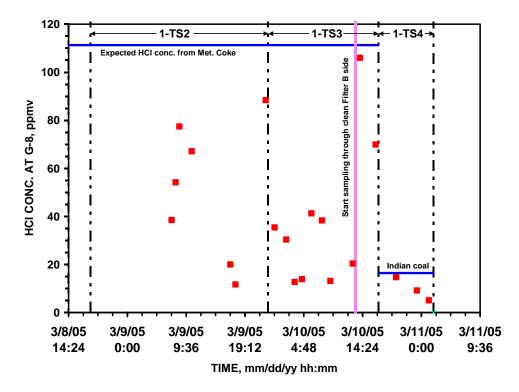


Figure 34 - Effect of Char on EPA Method 26 HCl Measurements When Switching from a Filter with Char (A Side) to a Cleaned Filter (B Side) on SE&C Skid #1

Prior to initiating trona injection, several HCl concentration measurements in the CF-R outlet syngas (i.e., at the G-13 sampling location) were made, to verify that the outlet HCl concentration was close to the inlet value. The highest HCl concentration measured was about 30 ppmv, before it was decided to proceed to the next test segment. It appeared some HCl removal was taking place in the CF-R vessel. This might be due to residual amounts of trona from pre-test sorbent feeder (T-2101) commissioning tests at pressure. Further evaluation of baseline results is required (particularly to determine whether any HCl is removed in the CF-R vessel by coke/coal char).

Figure 36 shows the measured HCl concentrations in the CF-R outlet syngas (i.e., at the G-13 sampling location) during trona injection (i.e., 1-TS3 test segment) as a function of test time and trona sorbent feed rate. Both time-averaged (batch) impinger measurements and on-line FT-IR analyses are shown. These measurements show good agreement and indicate that the HCl concentration slowly decreased during the trona injection test period. There appears to be a relationship between the trona feed rate and the HCl concentration in the CF-R product syngas: HCl concentration was further reduced as the trona feed rate was increased. The lowest HCl concentration in the CF-R outlet syngas was measured at 10.8 ppmv during trona injection (see sample ID # 051119-130 in Table 19). This represents about 90.3% HCl removal in the CF-R vessel.

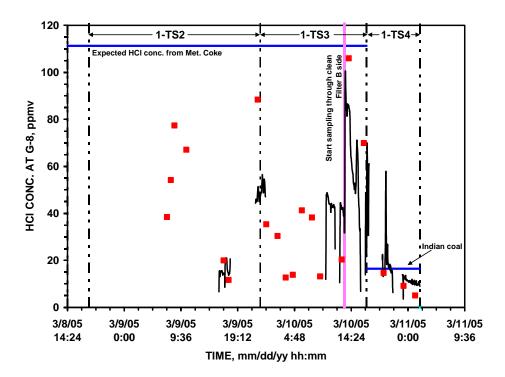


Figure 35 - Measured HCl Concentration in the Raw Syngas at Sampling Location G-8 (SE&C Skid #1)

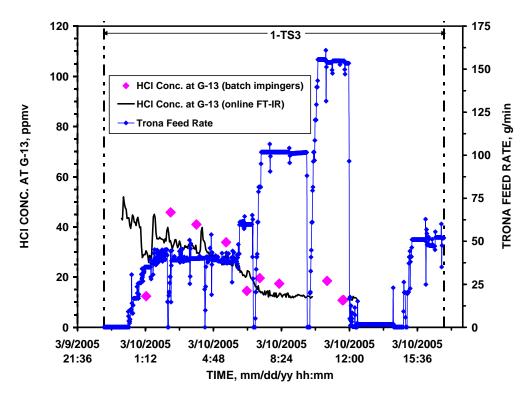


Figure 36 - Measured HCl Concentration in the CF-R Product Syngas at Sampling Location G-13 (SE&C Skid #2)

The trona feed rates shown in Figure 36 correspond to Na-to-Cl molar ratios (i.e., active sorbent component to contaminant) ranging from 5 to 30. These values are much higher than had been estimated in the Base Program (i.e., a Na-to-Cl molar ratio of 5 or less had been estimated to be sufficient to impart the desired HCl removal efficiency in the CF-R vessel). Several factors can cause this less than desired HCl removal performance. First, the temperature of the CF-R vessel (~ 800°F or 427°C) is lower than the optimum CF-R operating temperature of 932°F (500°C), which could have adversely affected the trona reactivity. Second, the water content of the raw syngas at the CF-R inlet (i.e., after the DSQ system) could have been too high leading to thermodynamic limitations. Third, the size distribution of the injected trona could be higher than desired, especially that, because metallurgical coke (much less reactive than coal) was used in this test campaign, the syngas flow rate was limited to about 1800 lbs/hr, which corresponds to a face velocity of about 2 ft/min (a face velocity > 3 ft/min was desired, as stated earlier).

A sample of the trona material used (Solvay T-200<sup>®</sup> sodium sesquicarbonate) was submitted for chemical and size distribution analyses to properly compare its performance with the results obtained in the Base Program with a naturally-occurring trona mineral from FMC in Green River, Wyoming. As shown in Table 16, the Solvay T-200<sup>®</sup> material assayed 31.6 Na compared to 35.6% for the FMC material used in the Base Program. This slight difference can be expected to lead to a lower effective capacity for the former material, but is not sufficiently significant to cause major reduction in performance. Similarly, although lower than the optimum temperature at which sorbent to contaminant ratios were estimated, the NGC-OPT1 operating temperature was not significantly lower than the lowest temperature investigated in the Base Program (i.e., 842°F or 450°C). At this lower temperature, trona was found to be more efficient in HCl removal (and at only slightly lower effective capacity) than was obtained at 500°C. Therefore, neither the nature of the bulk dechlorination material used nor the operating temperature of the CF-R filter-reactor could be expected to significantly influence the results obtained.

The steam content of the partially-cooled raw syngas at the CF-R inlet (i.e., at G-8) was estimated to range from 30 to 35 vol%, based on online measurements by the TF-IR instrument. To examine equilibrium limitations in the CF-R filter-reactor vessel, the estimated raw syngas composition was equilibrated with the trona active dechlorination component (i.e., Na<sub>2</sub>CO<sub>3</sub>) in the temperature range from 482°F (250°C) to 1202°F (650°C) with the steam content varied between 0 and 40 vol%. The results are summarized in Figure 37 and clearly indicate the equilibrium HCl concentration in the CF-R product syngas should be below 1 ppmv and therefore no equilibrium limitations exist under the operating conditions of the CF-R filter-reactor vessel.

The particle size distribution of the as-received Solvay T-200® is shown in Figure 38. For comparison, the Filter A char sample size distribution was also determined, as shown in Figure 39. The volume moment mean diameter (i.e., mean diameter with respect to the mass of the particles) is about 13  $\mu$ m and the median diameter, d(0.5), is about 8  $\mu$ m for the char sample. The corresponding values for the bulk dechlorination material were significantly higher, 53.8  $\mu$ m and 35.6  $\mu$ m, respectively. It appears likely that a large portion (at least half) of the trona fed would not have deposited on the filter elements given the relatively large portion of large particles in the trona, and the low syngas velocity through the Conditioning Filter-Reactor.

Figure 40 displays both the HCl concentration in the CF-R product syngas, as measured by the online FT-IR instrument at the G-13 sampling location, and the Conditioning Filter-Reactor pressure drop. As indicated earlier, during the trona injection test segment pulsing events took place every about 2 ½ hours. During the initial stages of the 1-TS3 segment, pulsing appears to have caused spikes in the measured HCl concentration. It should be noted the measured HCl concentration was influenced by the char accumulating on the Mott filter in the SE&C Skid #1, and so these spikes would not correspond to the inlet HCl value. The data shown in Figure 40 suggest these spikes appear to have diminished (or even eliminated) as more trona was fed. This trend supports the above analysis indicating that perhaps only a small portion of the trona injected reached the CF-R filter elements. The HCl concentration spikes might

have been eliminated once sufficient amounts of the very fine portions of trona accumulated on the candles.) Figure 41 shows a picture of the CF-R candles after completion of the first test campaign.

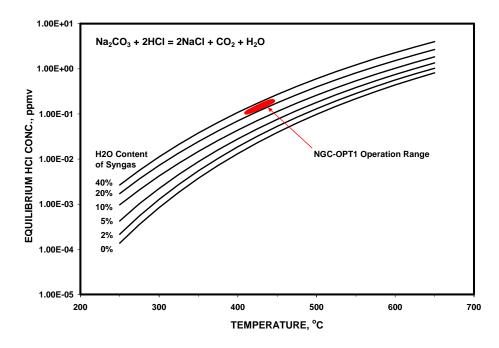


Figure 37 – Predicted Equilibrium HCl Concentration in the CF-R Product Syngas

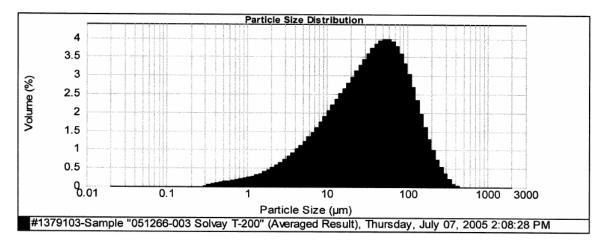


Figure 38 - Particle Size Distribution of the As-received Solvay T-200 Trona (Stage I Bulk Halide Sorbent)

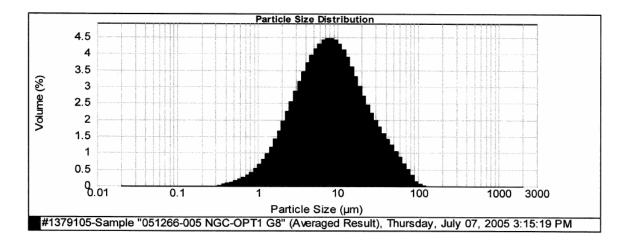


Figure 39 - Particle Size Distribution of the Syngas Ash Sample (Collected from Filter A on the SE&C Skid #1 at the G-8 Sampling Location)

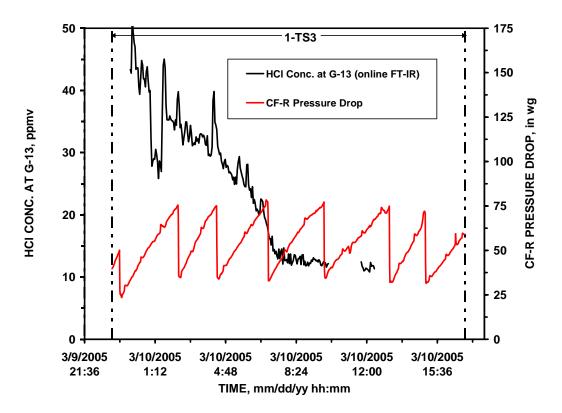


Figure 40 - Measured HCl Concentration in the CF-R Product Syngas and CF-R Pressure Drop during Trona Injection



Figure 41 – CF-R Candles (Post-Test)

### **Sulfur-Related Analytical Measurements**

The results from the gas chromatography analyses of the "dry" gas samples collected at the G-8 (CF-R inlet) and G-13 (CF-R outlet) sampling locations are summarized in Table 20. These results include the raw syngas bulk composition (i.e.,  $H_2$ ,  $CO_2$ ,  $N_2$ , CO,  $CH_4$ ) and total sulfur ( $H_2S$  and COS) analyses.

The H<sub>2</sub>S and COS measurements from the SE&C Skid #1 (dry sample) were evaluated. The metallurgical coke feed rate during the NGC-OPT1 test campaign varied between 336 and 350 lb/hr. The raw syngas (wet) flow rate was about 1850 lb/hr on the average (Table 18). The coke assayed 0.69% S. Assuming all coke sulfur is released during gasification, then the wet syngas would contain approximately 960 to 1,000 ppmv H<sub>2</sub>S. Assuming 32.5% H<sub>2</sub>O, the dry syngas sample might contain 1,422 to 1,482 ppmv. Coke conversion, however, was limited to < 50%. Therefore, it was possible to have 550 to 700 ppmv S as we measured ("grab" samples at G-8 by GTI CRS Lab, Table 20).

To confirm these estimates, selected samples from the T-502 vessel (S-5, Gasifier bottom ash) and the T-402 vessel (S-6, Secondary Cyclone fines) from samples taken between 04:00 and 17:20 on 3/9/2005 were analyzed for sulfur. During this test period, the total sulfur (H<sub>2</sub>S + COS) concentration in the dried syngas sample at G-8, as determined by GTI's CRS Lab, averaged ~ 660 ppmv, very close to the above estimate based on the coke sulfur content, operating conditions, carbon conversion, etc. The selected S-5 samples assayed 0.51% S. The selected S-6 samples assayed 0.56% S. Based on material balance considerations, the syngas H<sub>2</sub>S content was estimated to range from 500 to 650 ppmv. These analyses confirmed that the total sulfur released in the product gas correlated very well with coke conversion, and provided additional validation of our sampling and GC measurements.

Semi-continuous total sulfur ( $H_2S$  and COS) measurements using the HP 5890 GC with flame photometric detector (FPD) at the G-13 location (CF-R outlet) showed that COS concentration in the syngas was affected when trona was being injected. COS concentration decreased from the baseline level of about 65 ppmv to about 20 ppmv during trona injection. As shown in Figure 42, the COS level in the presence of trona was insensitive to the trona feed rate over the range from 40 to 150 g/min. This GC, with 15-minute cycle time, performed very well following transients on the time scale of the NGC Process. Results of sulfur measurements on batch samples corroborate this interesting observation, as can be seen in Table 20.

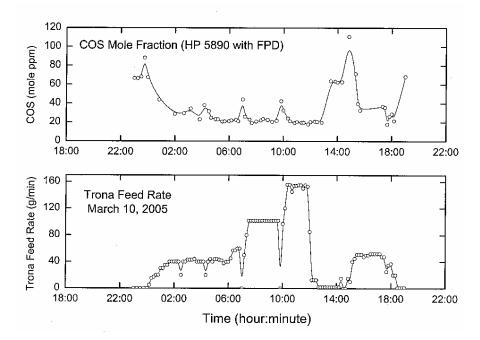


Figure 42 - Relationship between Trona Feed Rate and COS Concentration in the CF-R Outlet Syngas (Analysis by Prof. Peter Walsh, UAB)

During the NGC-OPT1 test campaign, some modifications were made to the SE&C Skid #2 (G-13). The original plan (see Figure 20) included maintaining the process gas at about 800°F as it passed through the Mott filter, and then reducing its temperature to just above 400°F in a heat traced heat exchanger with N<sub>2</sub> as the cooling medium. Tar from the syngas sample would be removed in a knockout pot below the heat exchanger. A grab sample would then be taken in a Silcosteel<sup>®</sup>-coated sample cylinder, in a similar arrangement to the SE&C Skid #1 (Figure 19), although this sample would contain steam. The canister is, however, not heated, and steam would later condense out in the cylinder. Pressure is reduced to about 40 psi before the gas goes through a Nafion dryer to perform warm temperature drying (presumably without affecting HCl). One sample of the dried gas is then sent to a GC and another (following further pressure reduction) to a set of impingers for HCl removal. Results from measurements with this approach are shown in Figure 43. The shaded region in Figure 43 covers sample results that were compromised by the condensation of water in the sample canisters. Laboratory analyses of H<sub>2</sub>S were erratic when liquid water from the samples was trapped in the GC sample column. However, excluding the compromised data, the results show a level of H<sub>2</sub>S in the process syngas that provides a good material balance for sulfur from the fuel and the unspent carbon in the gasification process, as discussed earlier.

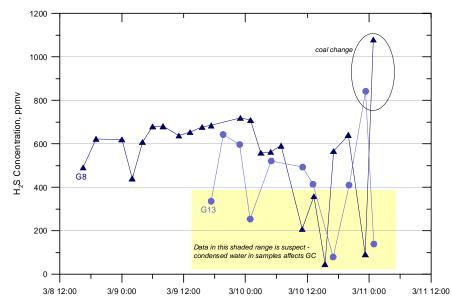


Figure 43 - Laboratory H<sub>2</sub>S Measurements from Sample Cylinders Collected at the SE&C Skids #1 and #2

The SE&C Skid #2 was modified significantly to deliberately condense the water from the process syngas by using cold water to chill the process gas to below laboratory temperatures. The "Hot Sample Line for Multiple Instrument Analysis" (Figure 20) was relocated upstream of the Mott filter. This hot line, which extends all the way into the Flex-Fuel second floor analytical station, was also used for providing samples to the sulfur GCs as well as the set of HCl impingers. Based on the sulfur analyses ("grab" samples) at the G-13 location, additional modifications to the SE&C Skid #2 were necessary for the NGC-OPT2 campaign to eliminate any water from the grab sample, which appears to be physically interfering with the GC measurements. The temperature controller on the heat exchanger was turned off and water (rather than nitrogen) was used as the cooling medium to control temperature to < 40°F, similar to the SE&C Skid #1. Similar modifications were also made to the SE&C Skid #3 at the G-19 location (ultra-clean syngas).

Ref	Lab Sample ID	Sample Point	Date	Time	Test	H2	CO2	O2/Ar	N2	со	CH4	Ethane	Ethene	H2S	COS
	-	-			Segment										
1	051119-010	G8	3/8/2005	16:30	1-TS1	5.5%	15.1%	0.81%	71.9%	6.48%	0.197%			492	108
2	051119-014	G8	3/8/2005	19:00	1-TS2	5.9%	14.4%	0.77%	71.8%	6.81%	0.213%			623	80
3	051119-029	G8	3/9/2005	0:00	1-TS2	5.6%	14.0%	0.74%	73.4%	6.07%	0.190%			620	74
4	051119-035	G8	3/9/2005	2:00	1-TS2	5.5%	14.2%	0.75%	73.6%	5.72%	0.190%			441	81
5	051119-044	G8	3/9/2005	4:00	1-TS2	5.2%	14.4%	0.75%	74.4%	5.02%	0.189%			608	65
6	051119-053	G8	3/9/2005	6:00	1-TS2	5.2%	14.4%	0.75%	74.5%	4.95%	0.193%			680	62
7	051119-056	G8	3/9/2005	8:00	1-TS2	5.1%	14.5%	0.75%	74.6%	4.81%	0.199%			681	58
8	051119-059	G8	3/9/2005	11:05	1-TS2	5.3%	14.5%	0.75%	74.2%	5.01%	0.195%			638	60
9	051119-070	G8	3/9/2005	13:15	1-TS2	5.5%	14.3%	0.75%	73.5%	5.61%	0.199%			654	65
10	051119-074	G8	3/9/2005	15:35	1-TS2	5.7%	14.3%	0.75%	73.0%	6.07%	0.202%			678	69
11	051119-075	G8	3/9/2005	17:20	1-TS2	5.9%	14.2%	0.75%	72.5%	6.43%	0.203%			685	69
12	051119-076	G13	3/9/2005	17:20	1-TS2	5.7%	13.8%	0.73%	73.0%	6.49%	0.199%			336	36
13	051119-082	G13	3/9/2005	19:40	1-TS2	5.5%	13.4%	0.72%	73.8%	6.32%	0.195%			643	34
14	051119-091	G13	3/9/2005	22:50	1-TS2	5.4%	13.9%	0.73%	73.9%	5.70%	0.208%			597	40
15	051119-092	G8	3/9/2005	23:00	1-TS2	5.7%	14.4%	0.74%	73.5%	5.29%	0.223%			719	60
16	051119-095	G8	3/10/2005	1:00	1-TS3	5.8%	14.2%	0.74%	73.1%	5.88%	0.213%			709	63
17	051119-096	G13	3/10/2005	0:55	1-TS3	5.2%	13.3%	0.70%	75.2%	5.44%	0.198%			254	32
18	051119-103	G13	3/10/2005	2:55	1-TS3	5.2%	12.9%	0.69%	75.3%	5.77%	0.193%			N/m	15
19	051119-104	G8	3/10/2005	3:00	1-TS3	5.9%	14.1%	0.74%	72.7%	6.32%	0.214%			559	65
20	051119-109	G8	3/10/2005	4:55	1-TS3	6.2%	13.9%	0.73%	72.3%	6.54%	0.223%			563	70
21	051119-110	G13	3/10/2005	5:00	1-TS3	5.5%	12.8%	0.69%	74.6%	6.12%	0.202%			521	16
22	051119-116	G8	3/10/2005	6:55	1-TS3	6.2%	13.7%	0.73%	72.1%	6.95%	0.221%			592	67
23	051119-117	G13	3/10/2005	7:00	1-TS3	5.6%	12.6%	0.68%	74.5%	6.47%	0.202%			N/m	N/m
24	051119-120	G13	3/10/2005	9:20	1-TS3	5.3%	12.7%	0.68%	75.0%	6.13%	0.199%			M/m	N/m
25	051119-126	G8	3/10/2005	11:00	1-TS3	5.9%	13.8%	0.73%	72.9%	6.41%	0.203%			208	58
26	051119-127	G13	3/10/2005	11:05	1-TS3	5.3%	12.7%	0.69%	74.9%	6.08%	0.193%			493	14
27	051119-133	G13	3/10/2005	13:05	1-TS3	5.3%	12.6%	0.69%	74.9%	6.22%	0.186%			414	12
28	051119-134	G8	3/10/2005	13:15	1-TS3	5.8%	13.8%	0.73%	73.1%	6.39%	0.198%			359	59
29	051119-141	G13	3/10/2005	15:20	1-TS3	4.9%	13.6%	0.72%	75.5%	5.18%	0.186%			N/m	51
30	051119-142	G8	3/10/2005	15:25	1-TS3	5.2%	14.3%	0.74%	74.3%	5.30%	0.194%			47	59
31	051119-144	G13	3/10/2005	17:00	1-TS3	4.8%	13.1%	0.70%	76.0%	5.21%	0.184%			79	18
32	051119-145	G8	3/10/2005	17:05	1-TS4	5.4%	14.2%	0.74%	73.9%	5.54%	0.201%			567	63
33	051119-161	G8	3/10/2005	19:55	1-TS4	5.4%	14.1%	0.75%	73.9%	5.53%	0.197%			641	65
34	051119-162	G13	3/10/2005	20:05	1-TS4	9.2%	13.4%	0.71%	67.6%	7.44%	1.559%			410	23
35	051119-102	G8	3/10/2005	23:15	1-134 1-TS4	9.9%	12.9%	0.72%	65.8%	8.68%	1.934%	0.003%	0.004%	92	54
36	051119-170	G13	3/10/2005	23:20	1-TS4	9.8%	12.8%	0.71%	65.8%	8.79%	1.939%	0.002%	0.004%	842	24
37	051119-176	G8	3/11/2005	0:50	1-134 1-TS4	9.8%	12.8%	0.71%	65.8%	8.84%	1.822%	0.002%	0.003%	1080	56
38	051119-170	G13	3/11/2005	0:55	1-134 1-TS4	9.7%	12.6%	0.72%	66.1%	8.97%	1.822%	0.002%	0.003%	139	25

 Table 20 - Summary of Batch Sample Gas Analyses during the NGC-OPT1 Test Campaign

# 11. NOVEL GAS CLEANING OPTIONAL PROGRAM TEST CAMPAIGN #2 (NGC-OPT2)

The second test campaign, NGC-OPT2, was performed as two separate campaigns, requiring two gasifier start-up periods in two different weeks (week of March 21 and week of March 28). Because of slight differences in gasification and NGC Process sections operating conditions, this test is treated as two separate tests, NGC-OPT2A and NGC-OPT2B.

### **11.1 Objectives**

The primary objective of the NGC-OPT2 was to evaluate the performance of the Test Filter-Reactor (TF-R) for sulfur (H<sub>2</sub>S and COS) and halide (HCl) removal (individually and simultaneously) to very stringent levels from syngas derived from the gasification of a carbonaceous feedstock. The Conditioning Filter-Reactor (CF-R) is to be operated under optimum operating conditions (830°F, 1-10 pulse cleaning frequency, and trona injected at optimum Na-to-Cl ratio when it is necessary to control HCl levels in the CF-R outlet). This was accomplished through a series of test segments, which were designed to:

- 1. set the Flex-Fuel gasifier operating conditions for producing syngas from the test feedstock (washed Indian coal) at 300 psig pressure, preferably with a syngas flow rate at the secondary cyclone exit of at least 3,300 lb/hr.
- 2. determine the operating conditions for the entire NGC Process "Conditioning" section to consistently result in a syngas with the target characteristics at the TF-R inlet: 1-5 ppmv total sulfur (H<sub>2</sub>S + COS), 1-5 ppmv halide (HCl), ~ < 0.1 ppmw particulate content, a temperature of 600 to 650°F (316 to 343°C), a pressure of ~ 200 psig, and a flow rate equivalent to a face velocity of at least 3 ft/min. The "Conditioning" section includes the Direct Spray Water Quench (DSQ) system for partial cooling of the raw syngas, the CF-R (including pulsing) vessel for particulate control, the Sorbent (Trona) Injection system for bulk HCl removal, a Sulfur Guard Bed (and associated pre-heater and slipstream syngas by-pass) for total sulfur control within the desired range at the TF-R inlet, and a Trim Cooler (indirect heat exchanger) for temperature control.</p>
- 3. operate the TF-R to confirm the feasibility of removing total reduced sulfur compounds (H<sub>2</sub>S and COS) and halide (mainly HCl) to < 60 ppbv and to < 10 ppbv, respectively, via selected dry, fine sorbent injection into the "conditioned" syngas upstream of the TF-R vessel. The key TF-R operating parameters include sorbent-to-contaminant ratio (i.e., sorbent feed rate), temperature, and pressure (i.e., face velocity).
- 4. confirm the effectiveness of "conventional" syngas Sample Extraction & Conditioning (SE&C) systems at Sample Points G-8 (CF-R inlet), G-13 (CF-R outlet), G-14 (conditioned syngas), and G-19 (ultra-cleaned syngas) with contaminant concentrations ranging from ppmv to ppbv levels.
- 5. provide "cold" and "hot/warm" slipstream samples from G-8, G-13, G-14, and G-19 for comprehensive, continuous, sensitive, and accurate measurements by personnel from the GTI Birmingham Office, as part of their project scope to develop an analytical suite as a key enabling technology for gasification systems integration.

### **11.2** Process Flow Diagram for NGC-OPT2

For this test series in the Flex-Fuel Test Facility (FFTF), the syngas was directed through the entire NGC Process section (Figure 6), which includes a "Conditioning" section and a "Test" section.

Syngas from the Secondary Cyclone (CY-401) is partially cooled in the DSQ system (HE-2001A) and then flows to the T-2153 CF-R. After passing through the filter elements or candles in the CF-R

vessel, the syngas is initially routed through the Pressure Let-Down station (PIC-602) to the Tertiary Cyclone (CY-601) and finally to the Flare (FL-601). During the Heat-Up period, after the Sulfur Guard Bed (R-2002) has been heated, the process gas flow is lined up from the T-2153 CF-R vessel through the Sulfur Guard Bed, the PCV-2006 pressure control valve, and the PIC-2181 pressure let-down valve to the Tertiary Cyclone (CY-601) and the Flare (FL-601). The T-2147 Test Filter-Reactor is bypassed, until the desired conditions are achieved in the "Conditioning" section of the NGC Process. After pulsing cycles are established with the nitrogen blow-back system on the T-2153 CF-R vessel, when it is necessary to control the HCl level in the CF-R outlet, trona (or nahcolite as an alternative halide sorbent), can be injected into the syngas stream, immediately before entering the T-2153 CF-R vessel, through the T-2101 Sorbent Injection system. The latter combines a loss-in-weight solids feeder with dilute-phase flow in nitrogen to convey the sorbent into the syngas.

Representative samples of the syngas are extracted, conditioned (de-dusted, cooled, and depressured), and analyzed with suitable instruments at two main locations:

- G-8: Partially-cooled raw syngas (between DSQ and CF-R)
- G-13: Partially-conditioned T-2153 CF-R product syngas (essentially dust-free, dechlorinated to 1-5 ppmv HCl, but still containing all the raw syngas sulfur).

As indicated above, the "Conditioning" section also comprises a Sulfur Guard Bed (SGB) and a Trim Cooler (indirect heat exchanger) for bulk sulfur removal and temperature reduction of the CF-R product gas to meet the requirements of the inlet syngas to the "Test" section. Once conditioned, the syngas stream is then introduced into the TF-R vessel for additional deep cleaning via selected dry, fine sorbents that are injected separately into the syngas immediately upstream of the TF-R vessel. Representative samples of the syngas are extracted, conditioned (de-dusted, cooled, and de-pressured), and analyzed with suitable instruments at two additional locations:

- G-14: Conditioned syngas (TF-R inlet)
- G-19: Ultra-cleaned syngas (TF-R outlet)

Other analytical work is also be performed on various samples to estimate CF-R particulate leakage (at S-13C), to characterize gasifier performance and efficiency in generating syngas from the test feedstock, to characterize process emissions for permitting activities, and to assess disposal options for the gasification and syngas cleanup byproduct materials. As shown in Figure 17, process samples include fuel feedstock, ash, dust, raw syngas, fresh sorbent materials, spent sorbents, and condensed liquids, in addition to several gas samples throughout the NGC Process section.

### 11.3 Run Sequence / Chronology

During the NGC-OPT2 campaign, the following sequence was followed to accomplish test objectives:

- 1. Conduct normal pressure testing to check for leaks throughout the Gasification and NGC Process "Conditioning" and "Test" sections.
  - 1.1. Gasifier pressure at 300 psig by PIC-2003; NGC Process "Test" section at 200 psig by PIC-2181
  - 1.2. Simulate / Practice: Cutting from T-2153 only into R-2002 etc. at 300 psig, By-passing T-2147 HSV-2047/HSV-2048
    - 1.2.1. Cutting from T-2153 only into R-2002 etc. while gasifying at 300 psig
    - 1.2.2. Cutting into T-2147 at 200 psig from by-pass mode, with HSV-2047/HSV-2049 system
- 2. Commission "Test" section Sorbent Feeders
  - 2.1. Establish N<sub>2</sub> flow rate versus differential pressure relationship for T-2107
  - 2.2. With injection N<sub>2</sub> at 15 scfm (FI-2107), verify ground, dried G-72E feeding at 3 g/min for WI-2107 (Stage II sulfur sorbent feeder)

- 2.3. Establish  $N_2$  flow rate versus differential pressure relationship for T-2108
- 2.4. With injection N<sub>2</sub> at 15 scfm (FI-2108), verify G-92C (or alternative sorbent/sorbent combination) feeding at 20 g/min for WI-2108 (Stage II halide sorbent feeder)
- 3. Verify SE&C skid viability at the G-8, G-13, G-14, and G-19 locations via spike-and-recovery methods (several HCl and S standards are available: 30 ppmv HCl/N<sub>2</sub>, 3 ppmv HCl/N<sub>2</sub>, 0.5 ppmv HCl/N<sub>2</sub>, 1200 ppmv H<sub>2</sub>S/N<sub>2</sub>, 0.5 ppmv H<sub>2</sub>S/N<sub>2</sub>). Impinger samples and analyzers. [Note: This is a major effort, and will need to be repeated during the test program to assure chemical stability and integrity of sampling systems.]
- 4. Heat up Gasification section and NGC Process "Conditioning" and "Test" sections
  - 4.1. Initiate pre-heating of Sulfur Guard Bed vessel (R-2002) with SH-2002 and LP nitrogen (up to 500°F)
    - 4.1.1. Heat-up gas to be routed through HE-2071 Bypass / T-2147 / PCV-2181 (Open 100%)
  - 4.2. Route Start-up Heater (SH-301) Flue Gas through HE-2001A initially, bypassing T-2153 and remainder of NGC Process section.
  - 4.3. When R-2002 temperature reaches 400°F, initiate heat up of gasifier with SH-301
  - 4.4. When SH-301 reaches Hi-Fire, route Flue Gas through T-2153 and remainder of NGC Process "Conditioning" section (everything except T-2147 TF-R vessel)
- 5. Calibrate Rosemount analyzer at the G-8 location (SE&C Skid #1); put it on line.
- Initiate second stage of heat up with metallurgical coke (Bethlehem Coke Breeze)
   Pressurize gasifier to 50 psig
- 7. Establish gasification with washed Indian coal
  - 7.1. Switch feed from metallurgical coke to washed Indian coal
  - 7.2. Establish gasifier bed level
  - 7.3. Increase gasifier pressure in stages to 200 psig, with PIC-602 system. Increase gasifier pressure in stages from 200 psig to 300 psig, with PIC-2006 system
  - 7.4. Adjust Gasifier operating parameters to achieve target gasifier pressure (300 psig) and target syngas flowrate (>3,300 lb/hr) operate gasifier at less than optimum carbon conversion to ensure long, uninterrupted operating durations for gasifier and the continuous generation of a suitable raw syngas for the NGC Process section for cleaning.
- 8. Establish preferred CF-R operating conditions (inlet temperature, outlet temperature)
  - 8.1. Observe pressure-drop increase on the T-2153 CF-R vessel and adjust pulsing as needed to give consistent "clean" differential pressure for filter candles
  - 8.2. Establish preferred conditions for CF-R pulsing frequency in the 1-10 range to handle dust loading from the washed Indian coal feedstock.
  - 8.3. Fine-tune pulsing on the T-2153 CF-R vessel to achieve cleaning at minimal nitrogen use
  - 8.4. Pulsing of the T-2153 filter with the fast-acting valves
  - 8.5. Pressure of Blow-Back system nitrogen in T-2304 Accumulator: starting point ~ twice process pressure (i.e., 400 to 450 psig)
  - 8.6. Length of pulsing time with fast-acting valves: 0.2 to 0.3 sec
  - 8.7. Note: An optimum temperature for the SGB is in the range 675 to 700°F (this should be a consideration in the operation of the CF-R; given that no HCl removal via trona injection is to be performed in this test, we have some flexibility to reduce the CF-R outlet temperature, if necessary, to match the desired SGB inlet temperature)
- 9. When reasonable syngas delivery conditions have been established, continue operating with washed Indian coal at "steady-state" conditions to verify/establish the following:

- 9.1. Measure HCl and total sulfur (H<sub>2</sub>S & COS) levels in the raw syngas (at G-8) and establish their variability (Note: HCl impinger measurements will require a clean Mott filter on SE&C Skid #1).
  - 9.1.1. Monitor raw gas composition at G-8 with mass spectrometer and GC/FPD/TCD. With G-13 hot-gas sample line to FT-IR inlet, monitor HCl concentration during pulsing on T-2153 to determine if nitrogen pulse affects measurement (can this be used to assess char contribution to HCl removal?).
- 9.2. Measure HCl levels with FT-IR and total sulfur (H<sub>2</sub>S & COS) in the raw syngas and CF-R product syngas (at G-8 and G-13)
  - 9.2.1. Throughout above test periods, monitor temperatures at the SGB inlet, SGB outlet, TC (Trim Cooler) inlet, TC outlet. Also, monitor syngas flow rate (FI-2005), quench temperature (TI-2022), etc.
- 9.3. If syngas temperature from T-2071 is too high, divert some syngas through HE-2071
  - 9.3.1. Initiate CWS through HE-2071 (Open Min Flow Bypass)
  - 9.3.2. Put TIC-2071 in Auto for 550°F
  - 9.3.3. Monitor CWR from HE-2071, to assure TI-2073 on CWR is less than 180°F
  - 9.3.4. With TIC-2006 in Manual, adjust process bypass around HE-2071 to give 600-650°F inlet to NGC Process "Test" section (TI-2073)
- 10. When preferred "Conditioning" section operating parameters are satisfied as described above:
  - 10.1. Establish syngas flow through T-2147 and establish process conditions for sorbent testing
    - 10.1.1. Check pressure on T-2147 -- should be 200 psig by back-pressuring
    - 10.1.2. Gradually open HSV-2047 and close HSV-2048, to establish syngas flow through T-2147 to PCV-2181
  - 10.2. Set conditions for sorbent testing
    - 10.2.1. Allow TF-R vessel to heat up (and achieve desired TF-R outlet temperature of ~ 550°F)
    - 10.2.2. Face velocity in T-2147 at Min of 3 ft/min (syngas rate at approx 3800 lb/hr
  - 10.3. Continue to operate NGC "Conditioning" section under optimum conditions to ensure TF-R desired inlet conditions for long, uninterrupted testing durations

Test Filter-Reactor
---------------------

Inlet temperature (°F):	600 to 650°F (after sorbent injection)
Outlet temperature (°F):	~ 550°F
Inlet gas flow (acfm):	> 123
Face velocity (ft/min):	> 3
Inlet Total Sulfur (ppmv):	1-5

- 10.3.1. Make frequent H<sub>2</sub>S (and COS) measurements, using online FPD GC.
- 10.3.2. If  $H_2S$  level too low, open Slipstream Bypass (FCV-2012) incrementally to give 1-5 ppmv  $H_2S$
- 10.4. Prepare to start operating the Stage II Halide Sorbent Feeder (T-2108) for deep HCl removal in the TF-R
  - 10.4.1. Set Transport Nitrogen rate (as controlled by differential pressure) required for adequate conveying of the fine G-92C sorbent into the "conditioned" process syngas
  - 10.4.2. Start with ~ 50 ft/sec injection velocity (15 scfm nitrogen rate (for 200 psig)), yielding approximately 30 in-H<sub>2</sub>O differential pressure.

- 10.4.3. Pre-coat filter candles with G-92C sorbent fines; feed G-92C in combination with nahcolite (50/50 mix by weight) at maximum feeding rate (about 100 g/min)
- 10.4.4. Reduce the Stage II halide sorbent (G-92C) injection rate to correspond to the maximum rate of Na/Cl=10 based on the syngas HCl content at G-14

G-92C (Süd-Chemie):	Chemical formula: Na <sub>2</sub> O supported on Al <sub>2</sub> O <sub>3</sub>
	Chemical analysis: 6.41% Na, 39.6% Al
	Approx. formula: 0.256 Na <sub>2</sub> O•0.744 Al <sub>2</sub> O <sub>3</sub>
	Bulk density: 45 lbs/cu ft (for 3/16-inch extrudates)
	Size distribution: ground at GTI to -325 mesh

G-92C Injection Rate (grams/min) =  $[(453.6)*(91.73)/[(0.512)*(60)*(10^{6})]]*[[HCl]_{o}*Q_{m}/MW_{sg}]*R_{Cl}$ 

- [HCl]<sub>o</sub> = Measured HCl concentration in conditioned syngas (at G-14), ppmv
- $Q_c = Conditioned syngas, lbs/hr$
- MW<sub>sg</sub> = Molecular weight of conditioned syngas, lb/lb-mole
- $R_{Cl} = Na$  to Cl molar ratio

Example: Raw syngas at 3,300 lbs/hr with an average MW of 25 and containing about 5 ppmv HCl, the G-92C sorbent injection required for a Na/Cl ratio of 10 is  $\sim$  8.9 g/min.

- 10.4.5. While allowing the system to stabilize (i.e., a somewhat constant fly ash/trona mixture composition of the filter cake), continue to measure total sulfur ( $H_2S + COS$ ) concentration and HCl levels with impingers in the TF-R inlet and outlet syngas (G-14 and G-19).
- 10.4.6. Monitor TF-R outlet gas composition at G-19 with GC/FPD/TCD/PFPD.
- 10.4.7. Attach G-19 hot-gas sample line to FT-IR inlet to monitor HCl concentrations.
- 10.4.8. If the TF-R product syngas HCl content (i.e., at G-19) is < 10 ppbv, continue operating at Na/Cl=10 for six (6) hours, which should result in at least 4-6 consistent samples. Note: If we do not achieve < 10 ppbv at G-19 with Na/Cl=10, then consider operating TF-R at a higher temperature or at a lower pressure (to increase face velocity)
- 10.4.9. Transition to a lower sorbent injection rate (i.e., Na/Cl=5) and measure effect of lower sorbent rate on HCl concentration in syngas with impingers at G-19 (3-4 hours or about 3 consistent samples).
- 10.4.10.Attach G-19 hot-gas sample line to FT-IR inlet to monitor HCl concentrations. Continue to monitor HCl concentration at G-14 (using FT-IR and/or impingers) and at G-19 (using impingers).
- 10.4.11.Shut off Stage II halide sorbent injection and Transport Nitrogen; allow system to stabilize (pulse clean manually to accelerate sorbent removal) and measure HCl in syngas with impingers at G-14 and G-19 (3 hours or 3 consistent samples)
- 10.5. Prepare to start operating the Stage II Sulfur Sorbent Feeder (T-2107) for deep sulfur removal in the TF-R
  - 10.5.1. Set Transport Nitrogen rate (as controlled by differential pressure) required for adequate conveying of the fine G-72E sorbent into the "conditioned" process syngas
  - 10.5.2. Start with ~ 50 ft/sec injection velocity (15 scfm (for 200 psig) nitrogen rate), yielding approximately 60 in-H<sub>2</sub>O differential pressure.

- 10.5.3. Pre-coat filter candles with G-72E sorbent fines; feed G-72E at maximum feeding rate (about 20 g/min, WI-2107)
- 10.5.4. Reduce the Stage II sulfur sorbent (G-72E) injection rate to correspond to the maximum rate of Zn/S=10 based on the syngas total sulfur (H<sub>2</sub>S + COS) content at G-14

G-72E (Süd-Chemie):	Chemical formula: ZnO supported on CaAl <sub>2</sub> O <sub>4</sub>
	Chemical analysis: 4.18% Al, 1.17% Ca, 69.8% Zn
	Approx. Formula: 0.928 ZnO•0.072 CaAl <sub>2</sub> O <sub>4</sub>
	Bulk density: 78 lb/cu ft (for 3/16-inch extrudates)
	Size distribution: ground at GTI to -325 mesh

G-72E Injection Rate (grams/min) =  $[(453.6)*(86.9)/[(0.928)*(60)*(10^6)]]*[[S]_0*Q_c/MW_{sg}]*R_s$ 

- [S]<sub>o</sub> = Measured total sulfur (H<sub>2</sub>S + COS) concentration in conditioned syngas (at G-14), ppmv
- $Q_c = Conditioned syngas, lb/hr$
- MW<sub>sg</sub> = Molecular weight of conditioned syngas, lbs/lb-mole
- $R_S = Zn$ -to-S molar ratio

Example: Raw syngas at 3,300 lbs/hr with an average MW of 25 and containing about 5 ppmv total S (H<sub>2</sub>S & COS), the G-72E sorbent injection required for a Zn/S ratio of 10 is  $\sim$  4.7 g/min.

- 10.5.5. While allowing the system to stabilize (i.e., a somewhat constant ash/sorbent mixture composition of the filter cake), continue to measure total sulfur ( $H_2S + COS$ ) concentration and HCl levels with impingers in the TF-R inlet and outlet syngas (G-14 and G-19).
- 10.5.6. Monitor TF-R outlet gas composition at G-19 with GC/FPD/TCD/PFPD.
- 10.5.7. Attach G-14 hot-gas sample line to FT-IR inlet to monitor HCl concentrations.
- 10.5.8. If the TF-R product syngas total S ( $H_2S \& COS$ ) content (i.e., at G-19) is < 60 ppbv, continue operating at Zn/S=10 for six (6) hours, which should result in at least 4-6 consistent samples. Note: If we do not achieve < 60 ppbv at G-19 with Zn/S=10, then we will consider other measures in consultation with Siemens (Dr. Newby) (operating at a higher temperature or at lower pressure (e.g., 100 psig) to increase face velocity)
- 10.5.9. Transition to a lower sorbent injection rate (i.e., Zn/S=5) and measure effect of lower sorbent rate on total S in syngas with PFPD GC and "grab" gas samples at G-19 (3-4 hours or about 3 consistent samples).
- 10.5.10. Attach G-19 hot-gas sample line to FT-IR inlet to monitor HCl concentrations. HCl sampling with impingers at both G-14 and G-19.
- 10.5.11.Shut off Stage II sulfur sorbent injection and Transport Nitrogen; allow system to stabilize (pulse clean manually to accelerate sorbent removal) and measure total S in syngas with PFPD and "grab" samples at G-14 and G-19 (3 hours or 3 consistent samples)
- 10.6. Prepare to start operating both the Stage II Sulfur and Halide Sorbent Feeders (T-2107 & T-2108) for simultaneous deep removal of total S (H<sub>2</sub>S & COS) and HCl in the TF-R
  - 10.6.1. Set Transport Nitrogen rate (as controlled by differential pressure) required for adequate conveying of the fine G-72E and G-92C sorbents into the "conditioned" process syngas
  - 10.6.2. Start with ~ 50 ft/sec injection velocity (15 scfm nitrogen rate), yielding approximately 60 in-H<sub>2</sub>O differential pressure.

- 10.6.3. Pre-coat filter candles with G-72E and G-92C sorbent fines; feed both G-72E and G-92C at maximum feeding rates
- 10.6.4. Reduce the Stage II sulfur and halide sorbents (G-72E and G-92C) injection rates to correspond to the maximum rates of Zn/S= 10 and Na/Cl=10 based on the syngas total S and HCl contents at G-14
- 10.6.5. While allowing the system to stabilize (i.e., a somewhat constant fly ash/sorbent mixture composition of the filter cake), continue to measure total sulfur ( $H_2S + COS$ ) concentration and HCl levels with impingers in the TF-R inlet and outlet syngas (G-14 and G-19).
- 10.6.6. Monitor TF-R outlet gas composition at G-19 with GC/FPD/TCD/PFPD.
- 10.6.7. If the TF-R product syngas (i.e., at G-19) total S content is < 60 ppbv and the HCl content is < 10 ppbv, continue operating at Zn/S=10 and Na/Cl=10 for six (6) hours, which should result in at least 4-6 consistent samples. Note: If we do not achieve < 10 ppbv HCl at G-19 with Na/Cl=10, then consider changing TF-R operating temperature or pressure (lower pressure to increase face velocity.)
- 10.6.8. Transition to lower sorbent injection rates (i.e., Zn/S=5 and Na/Cl=5) and measure effect of lower sorbent rates on total S and HCl concentrations in syngas with impingers and PFPD GC at G-19 (3-4 hours or about 3 consistent samples).
- 10.6.9. Shut off Stage II halide and sulfur sorbent injection and Transport Nitrogen; allow system to stabilize (pulse clean manually to accelerate sorbent removal) and measure HCl in syngas with impingers at G-14 and G-19 and total S concentration at both locations using PFPD GC (3 hours or 3 consistent samples)
- 11. Adjust Flex-Fuel gasifier operating conditions to optimize carbon conversion of the washed Indian coal feedstock
  - 11.1.1. Discontinue sending syngas through SGB, TC, and TF-R
  - 11.1.2. Route raw syngas through DSQ, through T-2153 CF-R, to pressure control valve(s), PCV-602, to Flare.
  - 11.1.3. Continue to operate the T-2153 CF-R (pulsing with no trona sorbent injection)
  - 11.1.4. Adjust Gasifier bed-ash to target level; achieve high-carbon conversion
  - 11.1.5. Adjust CF-R pulsing frequency as necessary
  - 11.1.6. Collect samples in the Gasification section more frequently for material and energy balances
  - 11.1.7. Measure HCl and (H<sub>2</sub>S & COS) at inlet (G-8) and outlet (G-13). Note: with the HCl content of the raw syngas expected to be  $\sim$  10 ppmv, no trona injection should be performed. The measurements taken will serve to relate contaminant concentrations in the raw syngas with the extent of carbon conversion.
  - 11.1.8. GC system samples to be collected from both G-8 and G-13 hot-gas sample lines.

### 11.4 Results and Discussion - NGC-OPT2

The Flex-Fuel Test Facility (gasification and Novel Gas Cleaning Process sections) was successfully operated throughout the NGC-OPT2A (week of March 21) and the NGC-OPT2B (week of March 28) test campaigns, including two Siemens' barrier filters (Conditioning Filter-Reactor, CF-R, and Test Filter-Reactor, TF-R), Sulfur Guard Bed, SGB (and associated low-pressure nitrogen pre-heater and slipstream by-pass system), and Stage II sulfur (G-72E) and halide (G-92C) sorbent feeders. The Trim

Cooler (located between the SGB and TF-R) was not utilized in these tests. In addition, finely-ground G-92C halide sorbent was fed as a 50:50 physical mixture with nahcolite to improve its feeding as determined in prior commissioning tests.

Because the raw syngas stream contains steam, it was necessary to heat up the SGB catalyst bed (Süd-Chemie's G-72E catalyst) in the NGC Process "Conditioning" section above the syngas dew point to avoid water condensation, which otherwise would severely damage the catalyst's desulfurization performance. Accordingly, during the NGC-OPT2A and NGC-OPT2B campaigns the raw syngas was initially routed from the DSQ system (HE-2001-A) through the CF-R vessel (T-2153), and then bypassed directly to the PCV-602 pressure let-down system. Simultaneously, low-pressure nitrogen was fed through the Nitrogen Pre-Heater (SH-2002) into the SGB vessel (R-2002), which had been isolated from the front-end of the NGC Process section (Figure 2 and Figure 31). The hot nitrogen exiting the SGB vessel was then routed around the Trim Cooler (HE-2071) through the Test Filter-Reactor (T-2147), and then through the PCV-2181 pressure let-down system. This made it possible to also pre-heat the TF-R vessel before directing the conditioned syngas stream into it.

Once the "Conditioning" section reached steady state and the TF-R brought to a sufficiently high temperature, the partially-cooled and essentially particulate-free syngas stream exiting the CF-R was sent through the SGB vessel, then bypassed through the PCV-2006, around the Trim Cooler, to the Condensate Knock-Out Tank (T-2072), and then to the TF-R. Pressure in the "Conditioning" section was controlled by the PCV-2003 pressure let-down system, and pressure in the "Test" section was controlled by the PCV-2181 pressure let-down system. In order to optimize the performance of the Stage II desulfurization and dechlorination sorbents in the "Test" section of the NGC Process, the temperature in the TF-R should be in the 550°F to 575°F range. Since no trona injection was necessary in the "Conditioning" section, the operating temperature for the CF-R vessel was determined primarily by the requirements of the SGB vessel. Therefore, the temperature strategy adopted involved (1) avoiding the complications of operating the HE-2071 cooler, with bypass, and (2) setting the inlet temperature to the CF-R vessel so that, with ambient heat losses from the intervening vessels and piping, the operating temperature of the TF-R vessel would be in the optimal range for sulfur and halide removal to the targeted ppbv levels.

During the second test campaign, raw syngas, derived from the gasification of washed Indian coal  $(0.56\% \text{ S}, < 100 \ \mu\text{g/g} \text{ Cl}, 0.14 \ \mu\text{g/g} \text{ Hg})$  and North Dakota lignite  $(0.89\% \text{ S}, ~ 122 \ \mu\text{g/g} \text{ Cl}, 0.06 \ \mu\text{g/g})$ Hg), was successfully conditioned to the temperatures (Table 21) and contaminant levels required at the inlet to the TF-R. Throughout testing, temperatures across the NGC Process section were maintained at ~ 1450°F at the secondary cyclone, ~ 1400°F at the inlet to the Direct Spray Water Quench, ~ 700°F at the inlet to the CF-R, ~ 650°F at the outlet of the CF-R, ~ 650°F at the SGB inlet, ~ 600°F at the SGB outlet,  $\sim 600^{\circ}$ F at the TF-R inlet, and  $\sim 550^{\circ}$ F at the TF-R outlet. These conditions ensured that the SGB was operated at optimal conditions for maximized utilization of the SGB catalyst and efficient sulfur removal. These conditions also ensured that contaminant removal reactions across the TF-R vessel were kept within the optimum temperature range (550°F to 575°F). The total sulfur concentration (H<sub>2</sub>S and COS) at the inlet was regulated within a 1-5 ppmv range throughout testing by maintaining a regulated syngas slipstream that bypassed the SGB vessel. An on-line GC/FPD provided near-continuous H<sub>2</sub>S and COS measurements (every 3 minutes). As indicated above, there was no need to engage the Trim Cooler for this test campaign. In addition, because of low levels of HCl in the raw syngas (~ 20 ppmv) and some measured HCl removal in the CF-R vessel (through interaction with char and residual trona) and in the SGB vessel (a small amount of CaO in the SGB catalyst), it was not necessary to engage the trona sorbent feeder upstream of the CF-R for bulk HCl removal. The measured HCl concentration in the syngas at the G-14 location (TF-R inlet) consistently averaged  $\sim 2$  ppmv. HCl measurements were made using both an on-line IMACC FT-IR instrument as well as by batch impinger sampling with ion chromatography (IC) analysis. Results from both test campaigns are discussed in more detail below.

	NGC-	OPT2A	NGC-OPT2B					
	Met Coke	Indian coal	Met Coke	Indian coal	Lignite			
		(washed)		(washed)				
Start	3/23/2005 at ~	3/23/2005 at ~	3/29/2005 at ~	3/29/2005 at ~	3/31/2005 at -			
	13:30	21:00	10:30	16:30	05:30			
End	3/23/2005 at ~	3/25/2005 at ~	3/29/2005 at ~	3/31/2005 at ~	3/31/2005 at -			
	21:00	05:00	16:30	05:30	15:00			
Duration, hr	7.5	32	6	37	9.5			
Gasifier								
Pressure, psig	50-200	200 - 255	50 - 125	125 - 250	250			
Bed Temp., °F	1750 - 1825	1625 - 1725	1750 - 1825	1675 - 1775	1550 - 1625			
Outlet Temp., °F	1730 1029 1775 - 1850	1600 - 1625	1775 - 1850	1075 - 1775	1550 - 1625			
Bed Height, ft	10 - 6	6 - 8	5 - 12	12 - 10	3 - 8			
Syngas Temp. at Secondary	-	1400 - 1475	-	1400 - 1525	1500 - 1350			
Cyclone, °F	_	1700 - 17/3	_	1700 - 1525	1500 - 1550			
Direct Spray Quench								
(DSQ)								
DSQ Inlet Temp., °F		1200 - 1475		1425 - 1525	1500 - 1350			
DSQ Outlet Temp., °F	-	800 - 650	-	725 - 750	725			
Syngas Flowrate, lbs/hr	-	2500 - 2900	-	2300 - 2900	2700 - 3000			
Pressure, psig	-	200 - 250	-	125 - 250	250			
Conditioning Filton								
Conditioning Filter- Reactor (CF-R)								
CF-R Inlet Temp., °F	-	700 - 710	-	725 - 750	725			
CF-R Outlet Temp., °F	-	650 - 670	-	675 - 700	675			
			1					
Sulfur Guard Bed (SGB)								
SGB Inlet Temp., °F	-	600 - 630	-	600 - 630	600 - 630			
SGB Outlet Temp., °F	-	600 - 630	-	600 - 630	600 - 630			
Trim Cooler	N/A	N/A	N/A	N/A	N/A			
	1V/ $A$	1N/A	1N/A	1N/A	1N/A			
Test Filter-Reactor (TF-								
R)								
TF-R Inlet Temp., °F		600 - 630		650 - 675	625 - 650			
TF-R Outlet Temp., °F		540 - 560		575 - 600	575			

# Table 21 - Gasification and NGC Process Sections Operating Conditions during the NGC-OPT2Test Campaign

# 11.4.1 NGC-OPT2A

Conditioning the raw syngas (at G-14, TF-R inlet) and deep halide removal by injection of finelyground G-92C sorbent (in combination with nahcolite) were the focus of the initial phase of the second test campaign (NGC-OPT2A). This campaign occurred during the week of March 21 and comprised the test segments shown in Table 22. As indicated, gasification of metallurgical coke (Bethlehem Coke Breeze, for second-stage heating up of the gasifier) and then washed Indian coal occurred over ~ 39 hours, from ~ 13:30 on 3/23/2005 through 05:00 on 3/25/2005. The period on washed Indian coal, during which NGC Process Stage II testing was performed, began at ~ 21:00 on 3/23/2005 and continued for 32 hours, until the test was terminated at ~ 05:00 on 3/25/2005. The gasification section was operated under steady state conditions throughout most of these test segments as demonstrated by the stable gasifier output shown in Figure 44. In this figure the hydrogen (H<sub>2</sub>), carbon monoxide (CO), and methane (CH<sub>4</sub>) concentrations in the raw, dry product syngas are given, as measured by the Rosemount CAT 200 online analyzer at the G-8 sampling location (CF-R inlet).

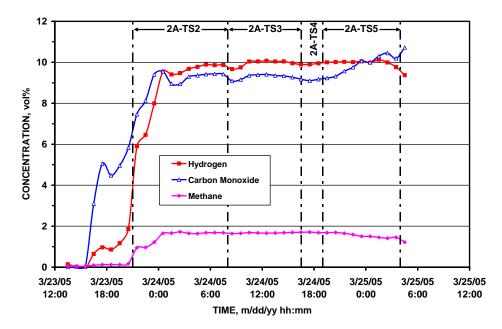


Figure 44 - H<sub>2</sub>, CO, and CH<sub>4</sub> in the Raw Gasifier Product Syngas at G-8 during the NGC-OPT2A Test Campaign

NGC-OPT2A		NGC-OPT2B	
	Indian coal (washed)		Indian coal (washed)
Overall		Overall	
Start	3/23/2005 at ~ 21:00	Start	3/29/2005 at ~ 16:30
End	3/25/2005 at ~ 05:00	End	3/31/2005 at ~ 05:30
Duration, hr	32	Duration, hr	37
Establishing Steady State with Indian coal (2A-TS2)		Sulfur sorbent injection (including TF-R "pre-coating"); 2B-TS3	
Start	3/23/2005 at ~ 21:00	Start	3/30/2005 at ~ 01:00
End	3/24/2005 at ~ 08:00	End	3/30/2005 at ~ 09:30
Duration, hr	11	Duration, hr	~ 8.5
		Pulse TF-R	3/30/2005 at ~ 11:00
Establishing Preferred Conditions in NGC Process Section (2A-TS3)		Simultaneous sulfur and halide sorbent injection (including TF-R "pre-coating"); 2B-TS5	
Start	3/24/2005 at ~ 08:00	Start	3/30/2005 at ~ 13:30
End	3/24/2005 at ~ 16:30	End	3/30/2005 at ~ 18:30
Duration, hr	8.5	Duration, hr	~ 5
<b>TF-R "pre-coating" with</b> halide sorbent mix (2A-TS4) Start	3/24/2005 at ~ 16:30	Breakthrough testing of	
Start	5/24/2005 at ~ 10.50	accumulated filter cake (from above test segment); 2B-TS6	
End	3/24/2005 at ~ 19:00	Start	3/30/2005 at ~ 18:30
Duration, hr	~ 2.5	End	3/31/2005 at ~ 05:00
		Duration, hr	~ 10.5
Halide sorbent injection (2A- TS5)			
Start	3/24/2005 at ~ 19:00	Transition to lignite (2B-TS7)	
End	3/25/2005 at ~ 04:00	Start	3/31/2005 at ~ 05:30
Duration, hr	~ 9	End	3/31/2005 at ~ 15:00
,		Duration, hr	~ 9.5
Terminate NGC-OPT2A	3/25/2005 at ~ 05:00		
		Pulse TF-R	3/31/2005 at ~ 10:30
		Simultaneous sulfur and halide	
		sorbent injection (including TF-R "pre-coating"); 2B-TS8	
		Start	3/31/2005 at ~ 12:15
		End	3/31/2005 at ~ 12:15 3/31/2005 at ~ 14:20
		Duration, hr	~ 2

 Table 22 - Test Segments in the NGC-OPT2 Test Campaign (March 21-25 & 28-31, 2005)

### 11.4.1.1 Gasifier and Filter-Reactor Operations

Temperature drop and pressure drop behaviors, and key process parameters (inlet syngas temperature, syngas mass flowrate, and operating face velocity) for the CF-R and TF-R vessels are reported in Figure 45 through Figure 48 for the NGC-OPT2A campaign. The figures shown cover the entire test duration, and on each figure the various test segments are delineated consistent with the information provided in Table 22. The Flex-Fuel gasifier pressure was brought up to ~ 250 psig after initiating the gasification of washed Indian coal (during test segment 2A-TS2). The inlet temperature for the syngas to the CF-R was also reduced from over 800°F to ~ 600°F and then raised back to ~ 650°F during this test segment. From about 08:00 on 3/24/2005 through the end of the gasification of Indian coal at 05:00 on 3/25/2005 the process conditions were relatively constant for the CF-R vessel.

The syngas mass flowrate at the CF-R inlet was quite stable, averaging ~ 2,740 lbs/hr, which corresponds to approximately 2.18 ft/min operating face velocity. The increase in syngas flowrate and face velocity shown on Figure 47 during the final test segment of the NGC-OPT2A campaign marks the beginning of our efforts to improve gasifier performance (i.e., increase carbon conversion). The solids loading in the syngas was moderate, and the time between pulses varied from about 22 minutes to 18-19 minutes at the end of the campaign. As shown in Figure 45, the differential pressure (PDI-2153) was allowed to increase to about 75 in wg before pulsing was initiated. After pulsing the differential pressure was about 20 in wg, indicating very effective pulsing with the fast-acting valves. The syngas temperature drop across the CF-R vessel appears to be in the range of 40 to 50°F, especially during steady state operating periods. This temperature drop is significantly lower than the one previously reported for the first test campaign (NGC-OPT1). This is due to the fact that the thermocouple on the syngas outlet from the CF-R vessel, TE-2155, was re-positioned during final preparations for this second test campaign. This new location is believed to indicate the actual syngas temperature more accurately than in previous tests involving this filter.

Figure 46 shows the measured temperature and pressure drop behavior for the TF-R vessel. Figure 48 shows the measured inlet syngas temperature and mass flowrate, and the calculated operating face velocity for the TF-R vessel. The syngas temperature at the TF-R inlet was reduced from over 745°F (at ~ 03:30 on 3/24/2005) and maintained at approximately 620°F throughout the 2A-TS3, 2A-TS4, and 2A-TS5 test segments. Temperature drop across the TF-R vessel ranged from ~ 65 to 75°F. Therefore, the TF-R was operated at a temperature approximating 550°F. The syngas mass flowrate at the TF-R inlet was stable, averaging ~ 2,480 lbs/hr, which corresponds to approximately 2.24 ft/min operating face velocity. During the 2A-TS4 test segment, when "pre-coating" of the TF-R candles was performed, the operating face velocity was ~ 2.5 ft/min, but steadily increased to slightly over 3 ft/min by the time sorbent injection was completed during the 2A-TS5 test segment.

The pressure drop behavior depicted in Figure 46 for the TF-R vessel is distinctly different from the pressure drop behavior for the CF-R vessel (Figure 45). Throughout the NGC-OPT2A campaign the TF-R was intentionally not pulsed (nor did it require pulsing). Prior to initiating sorbent injection during the 2A-TS4 test segment, i.e., from the time the test began to about 16:30 on 3/24/2005, the TF-R pressure drop was very stable at about 17 in wg. This is a very positive result because it clearly demonstrated there was no particulate leakage through the CF-R and that the syngas stream at the TF-R inlet was essentially particulate-free. During the candles "pre-coating" phase, i.e., 2A-TS4 test segment, which was performed for about 2.5 hours, the TF-R pressure drop increased steadily reaching about 80 in wg. This is also a very positive result because it clearly indicated that a portion of the fine sorbent mixture (finely-ground G-92C in combination with nahcolite) being injected was reaching the filter elements and uniformly depositing on their surfaces. As shown on Figure 46, when the sorbent feed rate was reduced to the test design rate during the 2A-TS5 test segment, the TF-R pressure drop continued to increase, but at a lower rate than in the previous test segment when the fine sorbent mixture was being injected at a much higher rate.

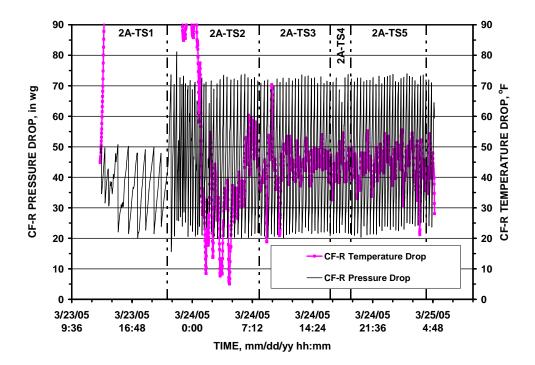


Figure 45 – Temperature and Pressure Drop Behavior of the Conditioning Filter-Reactor during the NGC-OPT2A Campaign

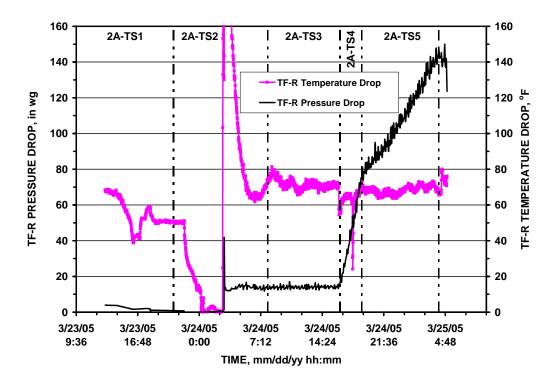


Figure 46 - Temperature and Pressure Drop Behavior of the Test Filter-Reactor during the NGC-OPT2A Campaign

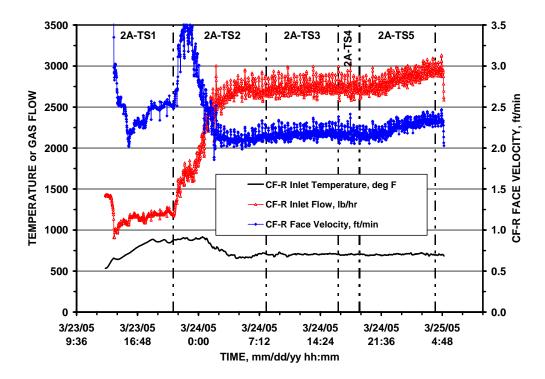


Figure 47 – Temperature, Syngas Flowrate, and Face Velocity at the Conditioning Filter-Reactor Inlet during the NGC-OPT2A Campaign

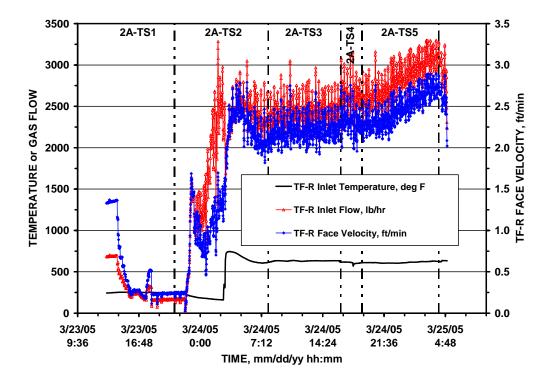


Figure 48 - Temperature, Syngas Flowrate, and Face Velocity at the Test Filter-Reactor Inlet during the NGC-OPT2A Campaign

### **11.4.1.2 Deep HCl Removal**

(During the one-week NGC-OPT2A campaign, efforts concentrated on controlling hydrogen chloride (HCl) in the TF-R and monitoring sulfur species concentrations throughout the NGC Process section. The fine, dry sorbent, a 50:50 physical mixture of nahcolite and finely-ground G-92-C, was injected from the T-2108 system into the conditioned syngas just upstream of the TF-R vessel. Commissioning tests with the preferred sorbent alone, i.e., G-92C, indicated the finely-ground material could not be injected consistently and without frequent intervention in the existing W-2108 K-Tron system. However, commissioning tests determined that nahcolite facilitated feeding of the finely-ground G-92C material.

Stage II halide sorbent injection was initiated at ~ 16:30 on 3/24/2005 and performed for ~ 12 hours during test segments 2A-TS4 and 2A-TS5, as indicated earlier in Table 22. Initially, the finely-ground G-92C/nahcolite mixture was injected at a much higher rate than was necessary (~ 100 g/min) to "pre-coat" the candle surfaces with sorbent materials and expedite testing. This test segment (2A-TS4) was continued for 2.5 hours, until the differential pressure ( $\Delta P$ ) across the TF-R reached ~ 75 in wg. Subsequently, the Stage II halide sorbent feed rate was reduced to the test design rate of 20 g/min and sorbent injection continued for the duration of the 2A-TS5 test segment. As shown in Figure 49,  $\Delta P$  across the filter increased steadily reaching ~ 150 in wg by the time this test phase was completed. This level of pressure drop was considered safe and the TF-R was conveniently not pulsed during this test campaign.

The results reported on Figure 49 clearly demonstrated that the injected material (or at least portions of it) reached the candles and uniformly deposited on their surfaces. The Stage II halide sorbent feed rate data does show some variability in the instantaneous rates, but the average rates (as determined from manual recordings of the K-Tron net weights at intervals of 30 to 60 minutes) were very close to the set points investigated (100 g/min and 20 g/min). Therefore, the T-2108 halide sorbent feeder functioned very well with the 50:50 mixture of the two materials.

Throughout testing, numerous samples were taken from a variety of locations and were analyzed using a wide array of instrumental methods. During Stage II halide sorbent injection, HCl concentrations were measured to be about 58, 61, and 52 ppbv (see Table 23, Lab Sample IDs 051143-074, -081, and 086). Table 23 provides a summary of all impinger/gas chromatography analyses made during the NGC-OPT2A test campaign at the G-8 (CF-R inlet), G-13 (CF-R outlet), G-14 (conditioned syngas at the TF-R inlet), and G-19 (ultra-cleaned syngas at the TF-R outlet) sampling locations. As indicated, each concentration at the G-19 location was measured from batch impinger samples that were obtained over a 2-hour sampling period. Using the approach described earlier for estimating the background HCl concentration and taking into account the total duration of syngas exposure during the NGC-OPT2B campaign, the time at which each sampling activity was performed, and whether or not test periods on metallurgical coke were considered, the background HCl concentration was estimated to range from ~ 40 to 55 ppbv. Therefore, the measurements made during the NGC-OPT2A campaign would represent approximately 3-18, 6-21, and 0-12 ppbv HCl in the ultra-clean syngas, as shown graphically in Figure 50. These HCl levels in the NGC Process Stage II product syngas are reasonably close to the desired output target of < 10 ppbv.

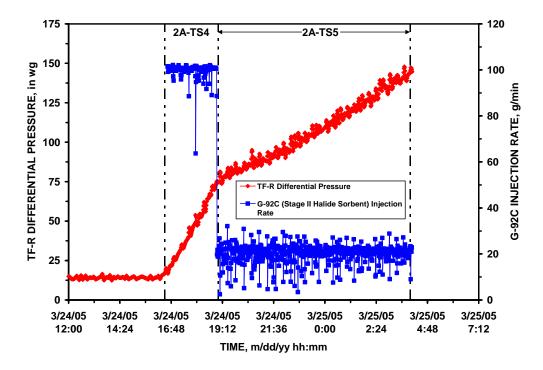


Figure 49 - Test Filter-Reactor Differential Pressure during Stage II Halide Sorbent Injection (NGC-OPT2A)

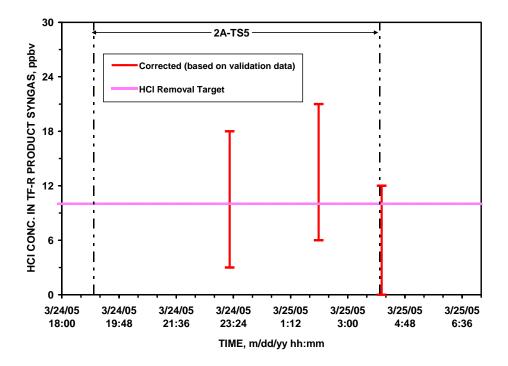


Figure 50 - Measured HCl Concentrations in the TF-R Product Syngas

The COS species was determined to be partially removed by the SGB material (see Table 24 and Table 26). It was also conclusively observed that the water-gas-shift reaction was taking place in the SGB vessel (a decrease in CO concentration accompanied by an increase in CO<sub>2</sub> and H<sub>2</sub> concentrations). For example, at the G-13 sampling point (upstream of SGB), gas analysis indicated 9.6% CO, 12.3% CO<sub>2</sub>, and 10% H<sub>2</sub> at ~ 11:45 on 3/24/05 (Lab Sample ID# 051143-041 in Table 24). At the G-14 sampling point (downstream of SGB), gas analysis indicated 6.21% CO, 15.4% CO<sub>2</sub>, and 12.9% H<sub>2</sub> at ~ 13:55 on 3/24/05 (Lab Sample ID# 051143-047 in Table 24). This is illustrated graphically in Figure 51.

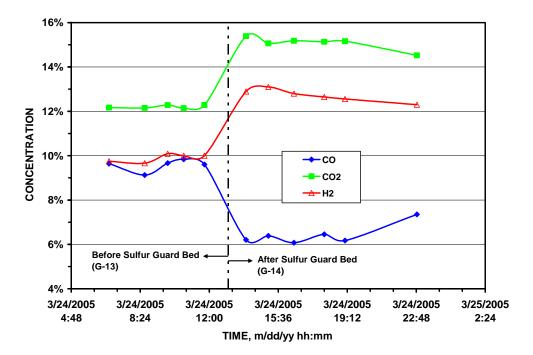


Figure 51 – CO, CO<sub>2</sub>, and H<sub>2</sub> Concentrations in the Syngas Upstream and Downstream of the Sulfur Guard Bed

Ref	Lab Sample ID	Sample Point	Date	Time Period	Average Time	Test	Measured HCI, ppmv
					(min)	Segment	
1	051143-006	G-14 Validation Test #1	03/24/05	00:16-00:51	35	2A-TS2	0.6
2	051143-007	G-14 Validation Test #2	03/24/05	00:56-01:24	30	2A-TS2	0.3
3	051143-016	G-8	3/24/2005	04:34-05:07	31	2A-TS2	<0.1
4	051143-017	G-13	3/24/2005	04:36-05:26	50	2A-TS2	4.2
5	051143-022	G-8	3/24/2005	05:59-06:31	32	2A-TS2	1.1
6	051143-023	G-13	3/24/2005	06:01-06:43	42	2A-TS2	2.3
7	051143-024	G-8	3/24/2005	07:19-07:51	32	2A-TS2	1.6
8	051143-025	G-13	3/24/2005	07:21-08:32	71	2A-TS3	1.8
9	051143-032	G-8	3/24/2005	09:39-10:05	26	2A-TS3	1.2
10	051143-037	G-8	3/24/2005	10:19-10:49	30	2A-TS3	1.3
11	051143-042	G-14	3/24/2005	10:34-11:04	30	2A-TS3	0.54
12	051143-043	G-14	3/24/2005	11:48-12:24	36	2A-TS3	0.35
13	051143-044	G-13	3/24/2005	12:30-13:09	39	2A-TS3	12.1
14	051143-045	G-13	3/24/2005	13:31-14:13	42	2A-TS3	14.4
15	051143-046	G-14	3/24/2005	13:32-14:13	41	2A-TS3	0.33
16	051143-051	G-13	3/24/2005	14:37-15:14	37	2A-TS3	14.2
17	051143-052	G-14	3/24/2005	14:37-15:14	37	2A-TS3	0.31
18	051143-056	G-14	3/24/2005	17:15-17:58	38	2A-TS4	0.37
19	051143-061	G-14	3/24/2005	18:04-19:04	60	2A-TS4	0.26
20	051143-064	G-14	3/24/2005	19:29-20:36	67	2A-TS5	0.27
21	051143-065	G-19	3/24/2005	20:13-22:17	64	2A-TS5	
22	051143-066	G-14	3/24/2005	21:42-22:44	62	2A-TS5	0.15
23	051143-074	G-19	03/24-03/25	22:20-00:13	53	2A-TS5	0.058
24	051143-075	G-14	03/24-03/25	23:18-00:27	69	2A-TS5	0.16
25	051143-080	G-14	3/25/2005	01:01-02:06	65	2A-TS5	0.11
26	051143-081	G-19	3/25/2005	01:05-03:05	120	2A-TS5	0.061
27	051143-083	G-14	3/25/2005	02:49-03:54	65	2A-TS5	0.095
28	051143-086	G-19	3/25/2005	03:09-04:58	109	2A-TS5	0.052
29	051143-087	G-14	3/25/2005	04:05-05:02	57		0.19
30	051143-088	G-14	3/25/2005	09:48-10:59	71		0.16
31	051143-089	G-19	3/25/2005	09:43-11:55	132		0.040
32	051143-090	G-14	3/25/2005	11:03-12:05	62		0.056
33	051143-099	G-19	3/25/2005	11:58-14:00	122		0.076

 Table 23 - Summary of Impinger/Ion Chromatography Analyses during the NGC-OPT2A Test Campaign

Ref	Lab Sample ID	Sample Point	Date	Time	Test	H2	CO2	O2/Ar	N2	СО	CH4	Hexane	H2S	COS
	_	-			Segment							Plus	(ppmv)	(ppmv)
1	051143-003	G-14 H2S Validation	3/23/2005	21:36	2A-TS2								1220	
2	051143-010	G-8	3/24/2005	2:10	2A-TS2	9.6%	12.4%	0.72%	65.9%	9.75%	1.556%	0.015%	959	83.5
3	051143-013	G-8	3/24/2005	3:40	2A-TS2	9.7%	12.3%	0.71%	65.9%	9.56%	1.731%	0.015%	300	70.9
4	051143-014	G-13 H2S Validation	3/24/2005	2:45	2A-TS2								195	l
5	051143-015	G-13 H2S Validation	3/24/2005	3:30	2A-TS2								449	
6	051143-020	G-8	3/24/2005	6:35	2A-TS2	9.8%	12.2%	0.70%	65.6%	9.63%	1.840%	0.020%	1041	68.7
7	051143-021	G-13	3/24/2005	6:46	2A-TS2	9.7%	12.2%	0.70%	65.9%	9.64%	1.763%	0.017%	167	52.9
8	051143-028	G-8	3/24/2005	7:55	2A-TS2	9.8%	12.3%	0.70%	65.7%	9.53%	1.808%	0.015%	992	57
9	051143-029	G-13	3/24/2005	8:38	2A-TS3	9.7%	12.2%	0.69%	66.5%	9.13%	1.795%	0.016%	814	57.5
10	051143-030	G-13	3/24/2005	9:50	2A-TS3	10.1%	12.3%	0.70%	65.4%	9.66%	1.854%	0.020%	322	47
11	051143-031	G-8	3/24/2005	10:10	2A-TS3	10.0%	12.3%	0.70%	65.5%	9.62%	1.786%	0.015%	583	74
12	051143-038	G-13	3/24/2005	10:40	2A-TS3	10.0%	12.1%	0.70%	65.5%	9.84%	1.746%	0.017%	569	51
13	051143-039	G-8	3/24/2005	11:00	2A-TS3	10.0%	12.2%	0.70%	65.6%	9.54%	1.798%	0.016%	646	70
14	051143-040	G-8	3/24/2005	11:55	2A-TS3	10.0%	12.2%	0.70%	65.6%	9.58%	1.768%	0.016%	854	68
15	051143-041	G-13	3/24/2005	11:45	2A-TS3	10.0%	12.3%	0.70%	65.6%	9.60%	1.759%	0.015%	700	52
16	051143-047	G-14	3/24/2005	13:55	2A-TS3	12.9%	15.4%	0.68%	63.1%	6.21%	1.718%	0.016%	6.52	0.08
17	051143-050	G-14	3/24/2005	15:05	2A-TS3	13.1%	15.1%	0.67%	63.0%	6.39%	1.747%	0.019%	1	0.05
18	051143-055	G-14	3/24/2005	16:25	2A-TS3	12.8%	15.2%	0.67%	63.5%	6.07%	1.728%	0.017%	1.09	0.06
19	051143-059	G-14	3/24/2005	18:00	2A-TS4	12.6%	15.1%	0.68%	63.3%	6.45%	1.735%	0.017%	122	3.5
20	051143-060	G-14	3/24/2005	19:05	2A-TS4	12.6%	15.2%	0.68%	63.6%	6.18%	1.782%	0.016%	1.7	0.23
21	051143-067	G-8	3/24/2005	22:33	2A-TS5	10.0%	12.3%	0.71%	65.3%	9.87%	1.750%	0.015%	1025	63.5
22	051143-068	G-14	3/24/2005	22:50	2A-TS5	12.3%	14.5%	0.69%	63.6%	7.35%	1.496%	0.010%	0.93	0.13
23	051143-071	G-14	3/25/2005	0:25	2A-TS5	12.4%	14.5%	0.68%	63.9%	7.05%	1.431%	0.009%	0.29	0.13
24	051143-076	G-14	3/25/2005	2:40	2A-TS5	13.0%	14.9%	0.69%	62.841%	7.139%	1.454%	0.010%	0.69	0.12
25	051143-077	G-8	3/25/2005	3:00	2A-TS5	10.0%	11.8%	0.72%	65.0%	10.91%	1.461%	0.010%	992	69.4
26	051143-082	G-14	3/25/2005	4:00	2A-TS5	12.4%	14.7%	0.69%	63.5%	7.17%	1.536%	0.011%	0.14	0.1

 Table 24 - Summary of Batch Sample Gas Analyses during the NGC-OPT2A Test Campaign

#### 11.4.2 NGC-OPT2B

The NGC-OPT2 test campaign was resumed on March 28, 2005 to accomplish the two remaining objectives: deep sulfur removal via injection of the finely-ground G-72E sorbent, followed by combined removal of HCl and sulfur species (H<sub>2</sub>S and COS) via simultaneous injection of both Stage II sorbents from two separate feeders. This one-week campaign comprised the test segments shown in Table 22. As indicated, gasification of metallurgical coke (Bethlehem Coke Breeze, for second-stage heating up of the gasifier), washed Indian coal, and then North Dakota lignite occurred over ~ 53 hours, from ~ 10:30 on 3/29/2005 to ~ 15:00 on 3/31/2005. The period on washed Indian coal, during which most of the NGC Process Stage II testing was performed, began at ~ 16:30 on 3/29/2005 and continued for ~ 37 hours, at which time lignite coal feeding was initiated. Testing with North Dakota lignite continued for ~ 10 hours, until the NGC-OPT2B campaign was terminated voluntarily at ~ 15:00 on 3/31/2005. The main goal of the testing with lignite was to confirm suitability of this feedstock for the third test campaign, focusing on mercury removal. However, deep sulfur and halide species removal was also evaluated during testing with lignite coal to confirm viability of the NGC Process with diverse carbonaceous feedstocks.

The gasification section was operated under steady state conditions throughout the NGC-OPT2B test segments as demonstrated by the stable gasifier output shown in Figure 52. In this figure the  $H_2$ , CO, and  $CH_4$  concentrations in the raw, dry product syngas are given, as measured by the Rosemount CAT 200 online analyzer at the G-8 sampling location (CF-R inlet).

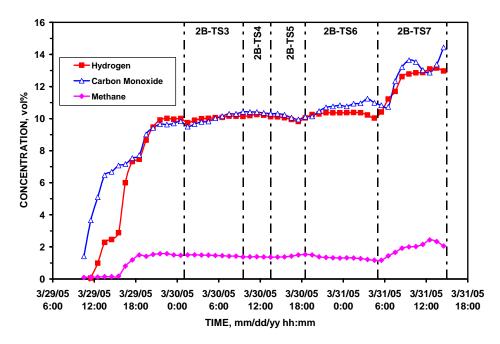


Figure 52 - H<sub>2</sub>, CO, and CH<sub>4</sub> in the Raw Gasifier Product Syngas at G-8 (CF-R Inlet) during NGC-OPT2B

### 11.4.2.1 Gasifier and Filter-Reactor Operations

Temperature drop and pressure drop behaviors, and key process parameters (inlet syngas temperature, syngas mass flowrate, and operating face velocity) for the CF-R and TF-R vessels are reported in Figure 53 through Figure 56 for the NGC-OPT2B campaign. The figures shown cover the entire test duration, and on each figure the various test segments are delineated consistent with the information provided in Table 22. The Flex-Fuel gasifier pressure was brought up to ~ 250 psig after initiating the gasification of washed Indian coal (during test segment 2B-TS2). The inlet temperature for

the syngas to the CF-R was also reduced from over  $875^{\circ}$ F (at ~ 15:00 on 3/29/2005) to ~ 725°F (at ~ 07:00 on 3/30/2005) and maintained at approximately this level for the remainder of this campaign. Figure 53 shows there were three distinct periods during which process conditions were relatively constant for the CF-R vessel: from ~ 19:00 on 3/29/2005 to 18:30 on 3/30/2005, from 18:30 on 3/30/2005 to 05:30 on 3/31/2005, and from 05:30 on 3/31/2005 to 15:00 on 3/31/2005 when the NGC-OPT2B campaign was terminated.

The Flex-Fuel gasifier performance (carbon conversion) was improved gradually over the NGC-OPT2B campaign, and accordingly, the syngas mass flowrate at the CF-R inlet increased steadily, as shown in Figure 55. The syngas mass flowrate increased from  $\sim 2,500$  lbs/hr during the 2B-TS2 test segment, when the preferred operating conditions for the gasifier were initially achieved, to slightly over 3,000 lbs/hr at the end of the 2B-TS6 test segment (i.e., end of operations with washed Indian coal). These syngas mass flowrates correspond to approximately 2 ft/min and 2.5 ft/min face velocity, respectively. During the transition from washed Indian coal to North Dakota lignite, the syngas mass flowrate and face velocity trends experienced a reduction, but eventually regained their previous values.

The solids loading in the syngas was moderate, and the time between pulses varied from about 15 to 18 minutes at the end of the campaign. As shown in Figure 53, the differential pressure (PDI-2153) was allowed to increase to about 60 in wg before pulsing was initiated. After pulsing the differential pressure was slightly over 20 in wg during operations with washed Indian coal up to  $\sim$  18:30 on 3/30/2005, and between 25 and 30 in wg during the 2B-TS6 (washed Indian coal) and 2B-TS7 (North Dakota lignite) test segments. CF-R pulsing was performed effectively throughout the NGC-OPT2B campaign. The syngas temperature drop across the CF-R vessel initially varied between 40 and 60°F, but appears to have stabilized in the 40 to 50°F range, once the syngas temperature at the CF-R inlet was controlled to  $\sim$  725°F. This temperature drop is quite similar to the one previously reported for the first phase of this test campaign (NGC-OPT2A).

Figure 54 shows the measured temperature and pressure drop behavior for the TF-R vessel. Figure 56 shows the measured inlet syngas temperature and mass flowrate, and the calculated operating face velocity for the TF-R vessel. The syngas temperature at the TF-R inlet was reduced from ~ 725°F (at ~ 05:00 on 3/30/2005) and maintained at approximately 639°F, 672°F, and 642°F throughout the 2B-TS5, 2B-TS6, and 2B-TS7 test segments. Temperature drop across the TF-R vessel ranged from ~ 65 to 75°F. Therefore, the TF-R was operated at a temperature approximating 550°F. The syngas mass flowrate at the TF-R inlet and the face velocity, shown in Figure 56, exhibit similar trends to their counterparts at the CF-R inlet, shown in Figure 55. The syngas mass flow rate increased steadily over the test period, from about 2,500 lbs/hr to over 3,250 lbs/hr. Correspondingly, the TF-R face velocity increased from about 2 ft/min to over 3.2 ft/min.

The pressure drop behavior depicted in Figure 54 for the TF-R vessel is distinctly different from the pressure drop behavior for the CF-R vessel (Figure 53). Prior to initiating sorbent injection during the 2B-TS3 test segment, the TF-R candles were pulsed to remove the accumulated G-92C/nahcolite sorbent mixture from the previous campaign (NGC-OPT2A). As shown on Figure 54, from ~ 19:00 on 3/29/2005 to ~ 01:00 on 3/30/2005, the TF-R pressure drop was very stable at about 32 in wg. This is a very positive result because it clearly demonstrated there was no particulate leakage through the CF-R and that the syngas stream at the TF-R inlet was essentially particulate-free. During the candles "pre-coating" phase, which was performed for about 1.5 hours within the 2B-TS3 test segment, the TF-R pressure drop increased steadily reaching about 95 in wg. This is also a very positive result because it clearly indicated that a portion of the fine sorbent mixture (finely-ground G-72E) being injected was reaching the filter elements and uniformly depositing on their surfaces. As shown on Figure 54, when the sorbent feed rate was reduced to the test design rate during the 2B-TS3 test segment, the TF-R pressure drop continued to increase, but at a lower rate. Figure 54 reveals that the  $\Delta P$  was recovered back to about 30 in wg

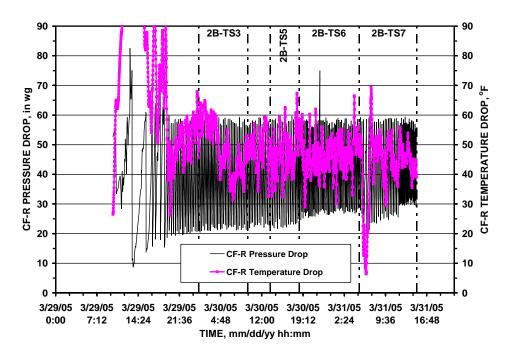


Figure 53 - Temperature and Pressure Drop Behavior of the Conditioning Filter-Reactor during the NGC-OPT2B Campaign

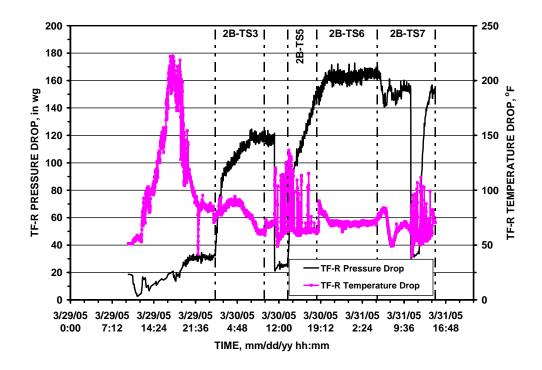


Figure 54 - Temperature and Pressure Drop Behavior of the Test Filter-Reactor during the NGC-OPT2B Campaign

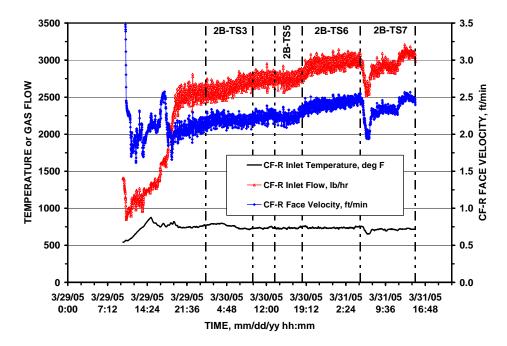


Figure 55 - Temperature, Syngas Flowrate, and Face Velocity at the Conditioning Filter-Reactor Inlet during the NGC-OPT2B Campaign

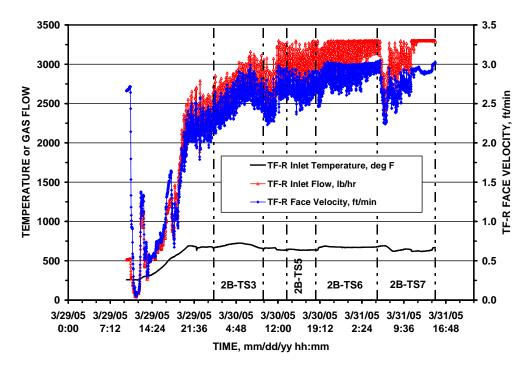


Figure 56 - Temperature, Syngas Flowrate, and Face Velocity at the Test Filter-Reactor Inlet during the NGC-OPT2B Campaign

following pulsing of the TF-R elements at ~ 11:00 on 3/30/2005 once the 2B-TS3 test segment objectives were achieved. This figure also shows the TF-R pressure drop behavior during the remaining test segments, including sorbent injection for simultaneous sulfur and HCl control, breakthrough testing of accumulated filter cake, and sorbent injection during operation with lignite coal. Details on these test segments are provided below, in connection with presentation and discussion of the measured cleaning efficiencies via dry sorbent injection.

# 11.4.2.2 Deep Sulfur Removal

Once the gasification and NGC Process sections operating conditions were established, the Test Filter-Reactor was pulsed. The H<sub>2</sub>S concentration in the conditioned syngas at the TF-R inlet (i.e., G-14 sampling location) was increased to the target level of 1 to 5 ppmv, by opening up a slight flow in the slip-stream bypass around the R-2002 SGB vessel through the FCV-2012 system. The sulfur removal test segment (2B-TS3) was then started at ~ 01:00 on 3/30/2005 with pre-coating the filter elements (candles) by feeding the finely-ground G-72E sorbent, using the T-2107 Stage II Sulfur Sorbent Feeder, at the higher rate of 30 g/min. The pressure differential ( $\Delta P$ ) across the TF-R vessel, initially at ~ 30 in wg, began to increase significantly, and after 1.5 hours the TF-R  $\Delta P$  reached ~ 95 in wg. The Stage II sulfur sorbent feed rate was then reduced to 5 g/min. As in the previous halide sorbent injection test completed in the prior week,  $\Delta P$  across the TF-R continued to increase steadily, but at a slower rate than during the "pre-coating" phase. At the conclusion of the 2B-TS3 test segment, when sorbent injection was terminated, the TF-R  $\Delta P$  stabilized and did not increase any further.

During this campaign, the total sulfur ( $H_2S + COS$ ) level was maintained at ~ 2-5 ppmv at the TF-R inlet (G-14) through regulating the amount of raw syngas that was bypassed around the SGB vessel. Figure 57 reports the  $H_2S$  concentration in the conditioned syngas at the G-14 sampling location, as measured by the on-line GC/FPD (located in the FFTF second floor analytical station), during the Stage II sulfur sorbent injection test segment (2B-TS3). As shown, the  $H_2S$  concentration was maintained in the 2-5 ppmv range throughout this test segment. Figure 57 also shows that the total sulfur concentration measured at the TF-R product gas (i.e., G-19 sampling location), using batch samples collected at the SE&C Skid #3 was consistently below the 50 ppbv detection limit of GTI Analytical Laboratory's instrument. In Figure 57, the numerous total sulfur determinations made are shown arbitrarily at the 25 ppbv level.

Figure 57 shows that total sulfur concentration in the ultra-cleaned syngas measured below 50 ppbv also throughout the 2B-TS5 test segment, during which simultaneous control of sulfur and halide species was performed via dry sorbent injection of the finely-ground G-92C and G-72E sorbents (from two separate feeders).

# 11.4.2.3 Simultaneous Sulfur and Halide Species Removal

Following the sulfur removal test segment (2B-TS3), the Test Filter-Reactor was pulsed at ~ 11:00 on 3/30/2005 (to dislodge the accumulated filter cake of essentially Stage II sulfur sorbent) and readied for the next test segment: the combined removal of sulfur and halide species (H<sub>2</sub>S, COS, HCl). A "pre-coating" phase was first performed with the G-92C/nahcolite mixture (from the T-2108 Stage II Halide Sorbent Feeder) at 50 g/min and the G-72E sorbent (from the T-2107 Stage II Sulfur Sorbent Feeder) at 20 g/min. The pressure differential on the T-2147 TF-R began to increase significantly and within an hour,  $\Delta P$  across the TF-R increased to ~ 95 in wg. As the sorbent injection rates were reduced at ~ 14:30 to the test target rates of about 10 g/min for the Stage II sulfur sorbent and 20 g/min for the Stage II halide sorbent (in combination with nahcolite), the rate of increase in the TF-R pressure differential decreased. Combined sulfur and halide contaminant removal continued for approximately 5 hours, when  $\Delta P$  across the TF-R reached 150 in wg. When sorbent injection was terminated at ~ 18:30, the pressure differential stabilized and did not increase any further.

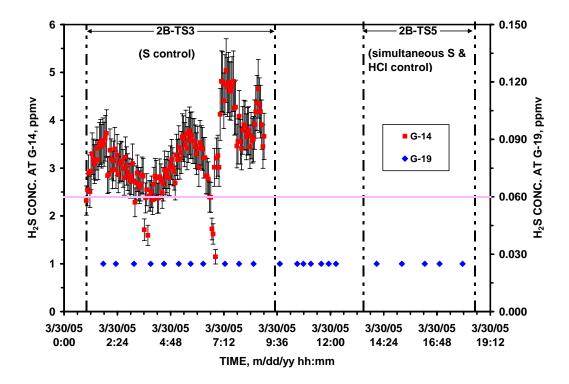


Figure 57 – Measured H<sub>2</sub>S Concentration at the Test Filter-Reactor Inlet (Conditioned Syngas at G-14) and Outlet (Ultra-cleaned Syngas at G-19)

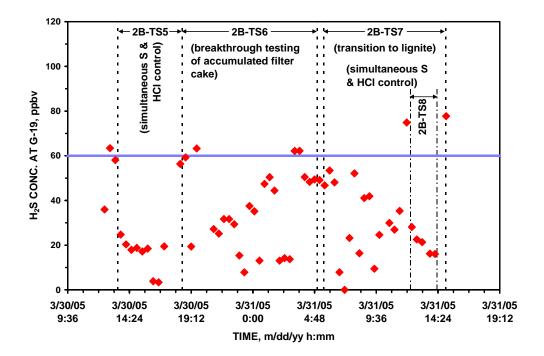


Figure 58 - H<sub>2</sub>S Concentration in the Ultra-Clean Syngas at the Test Filter-Reactor Outlet

As indicated earlier, the total sulfur concentration ( $H_2S$  and COS) in the TF-R outlet (at G-19) was measured consistently at less than 50 ppbv, using batch samples. In addition, as shown in Figure 58, semi-continuous online sulfur measurements (every 24 minutes) using the Varian CP-3800 PFPD gas chromatograph were quite stable, averaging ~ 20 ppbv, well within our desired output level of < 60 ppbv.

### 11.4.2.4 "Breakthrough" Testing of Accumulated Filter Cake

At the conclusion of the 2B-TS5 (simultaneous removal of sulfur and halide species) test segment, with sorbent injection being stopped, very low contaminant concentrations continued to be measured in the TF-R outlet (G-19 sampling location). Therefore, instead of pulsing the TF-R, it was decided to continue to monitor contaminant concentrations at the inlet and outlet of the TF-R for an extended period of time, to evaluate the effectiveness of the accumulated filter cake for continued removal of sulfur and halide species to the desired output levels. Gasifier operations were continued with washed Indian coal. With H2S breakthrough still not detected after more than 4 hours of such operation, the total sulfur concentration at G-14 (the TF-R inlet) was raised (by increasing the flowrate through the slipstream bypass around the SGB vessel) to  $\sim 25$  ppmv, as shown in Figure 59. To develop additional insights into the nature, amount, uniformity, etc. of the filter cake on the TF-R candles, this "breakthrough" testing of accumulated filter cake (i.e., 2B-TS6 test segment on Figure 59) continued overnight. The outlet total sulfur concentration was monitored overnight, and as shown in Figure 58, throughout this period, the total sulfur concentration ranged between 20 and 40 ppby, indicating the efficacy of the Test Filter-Reactor in removing sulfur and the flexibility of the NGC Process with respect to regulating the sulfur level at the Stage II inlet. Thus, it appears that sulfur levels at the TF-R inlet do not need to be maintained within a strict 1-5 ppmv range to achieve a desired output sulfur level of < 60ppbv.

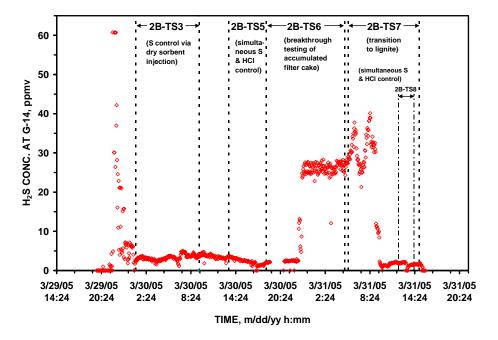


Figure 59 - H<sub>2</sub>S Concentration in the Conditioned Syngas at the Test Filter-Reactor Inlet during the NGC-OPT2B Test Campaign

### 11.4.2.5 NGC Process Stage II Testing with Lignite-Derived Syngas

At approximately 06:30 on 3/31/05, we transitioned into lignite as a feedstock from washed Indian coal. Once stable operating conditions were established, the TF-R vessel was pulsed at  $\sim 10:30$  on 3/31/05, to dislodge the TF-R filter cake (G-72E/G-92C/nahcolite mixture). More sorbent quantities were added into the two Stage II sorbent feeders and combined sulfur and halide removal via sorbent injection was performed. As sorbent injection from both the T-2107 and T-2108 systems was commenced at ~ 12:13 on 3/31/05, the pressure differential began to increase significantly on the TF-R. Initially, the rate of sorbent addition was 30 g/min for the Stage II sulfur sorbent (from the T-2107 system) and 50 g/min for the Stage II halide sorbent (in combination with nahcolite, from the T-2108 system). At  $\sim$  12:26, the rate of the sulfur sorbent addition was decreased to 25 g/min, and at ~ 12:59 the set-point was further reduced to 20 g/min, and then to 15 g/min at  $\sim$  13:08. As the sorbent injection rates were reduced at  $\sim$ 13:14 from the "pre-coat" rates to the test target rates (10 g/min for T-2107 and 20 g/min for T-2108), the rate of increase in the T-2147 pressure differential decreased. When sorbent injection was terminated at about  $\sim$  14:20, the pressure differential stabilized and did not increase any further. As shown in Figure 58 and Table 26, the same cleaning efficiencies were measured during this test (with lignite) as were achieved with the washed Indian coal, showing that the NGC Process properly functions with two diverse fuels.

The particle size distribution (PSD) of the Stage II halide sorbent (i.e., finely-ground G-92C) is shown in Figure 60 and that for the Stage II sulfur sorbent (i.e., finely-ground G-72E) is shown in Figure 61. The volume moment mean diameter (i.e., mean diameter with respect to the mass of the particles) is about 13.5  $\mu$ m and the median diameter, d(0.5), is about 8.2  $\mu$ m for the dechlorination sorbent. The corresponding values for the desulfurization sorbent were lower, 6.5  $\mu$ m and 3.5  $\mu$ m, respectively. Size distributions for the Stage II sorbents are similar or lower than that for the char sample. The corresponding values for the Solvay sodium bicarbonate material (nahcolite), which was used to facilitate feeding of the finely-ground G-92C material, are about 64  $\mu$ m and 60  $\mu$ m, respectively.

The fine particle sizes of the Stage II sulfur and halide sorbents used appear to have worked very well. Additional work is needed to optimize Stage II sorbent particle sizes and size distributions to determine optimum size ranges to facilitate sorbent feeding and entrainment, and to result in a filter cake with optimum properties. Smaller sorbent particles may result in higher filter cake pressure drops and lower filter cake permeability, leading to either limited filter face velocities or more frequent pulse cleaning. Smaller sorbent particles may also result in difficulties in sorbent feeding, since they may not be free-flowing and may agglomerate and/or easily bridge feed hoppers. They may also result in filter cake "sintering" in some cases. The optimum mean diameter of the sorbent particles should probably be no less than 10 µm and could be as large as 20 µm.

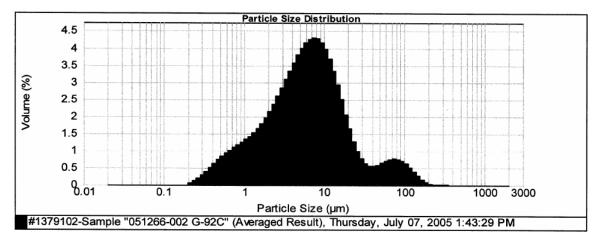


Figure 60 - Particle Size Distribution of the Finely-Ground G-92C Stage II Halide Sorbent

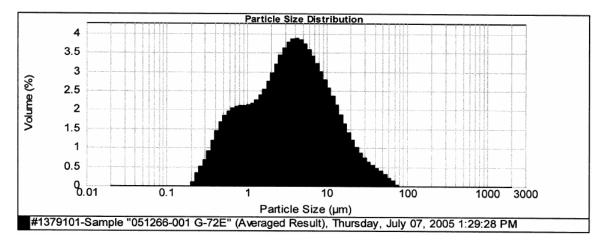


Figure 61 - Particle Size Distribution of the Finely-Ground G-72E Stage II Sulfur Sorbent

Ref	Lab Sample ID	Sample Point	Date	Time Period	Average Time (min)	Test Segment	Measured HCI, ppmv
34	051143-100	G-14 Blank #1	3/29/2005	14:55-16:48	113	2B-TS2	0.028
35	051143-101	G-19 Blank # 1	3/29/2005	15:00-17:47	167	2B-TS2	< 0.020
36	051143-106	G-19 Blank # 2	3/29/2005	17:52-20:31	159	2B-TS2	0.023
37	051143-112	G-19 Blank # 3	3/29/2005	21:15-23:11	116	2B-TS2	< 0.020
38	051143-132	G-19	3/30/2005	00:52-03:48	176	2B-TS3	0.036
39	051143-141	G-19	3/30/2005	03:53-06:06	133	2B-TS3	0.066
40	051143-142	G-14	3/30/2005	03:48-06:38	170	2B-TS3	0.130
41	051143-148	G-19	3/30/2005	07:02-10:04	182	2B-TS3	0.048
42	051143-152	G-14	3/30/2005	07:06-09:43	157	2B-TS3	0.079
43	051143-163	G-14	3/30/2005	10:10-13:08	178	2B-TS4	0.049
44	051143-164	G-19	3/30/2005	10:15-13:13	178	2B-TS4	0.033
45	051143-180	G-14	3/30/2005	14:02-16:40	158	2B-TS5	0.060
46	051143-181	G-19	3/30/2005	14:06-15:56	164	2B-TS5	0.100
47	051143-186	G-19	3/30/2005	16:04-17:55	111	2B-TS5	0.059
48	051143-195	G-19	3/30/2005	18:00-20:11	131	2B-TS5	0.065
49	051143-196	G-14	3/30/2005	18:06-20:42	156	2B-TS5	0.034
50	051143-201	G-19	3/30/2005	20:28-22:38		2B-TS6	0.076
51	051143-209	G-19	3/31/2005	23:49-01:??		2B-TS6	0.041
52	051143-214	G-19	3/31/2005	02:23-04:26		2B-TS6	0.035
53	051143-233	G-13	3/31/2005	11:38-13:20		2B-TS7	2.030
54	051143-237	G-19	3/31/2005	12:50-14:00		2B-TS7	0.029
55	051143-238	G-8	3/31/2005	13:30-14:00		2B-TS7	0.250

 Table 25 - Summary of Impinger/Ion Chromatography Analyses during the NGC-OPT2B Test Campaign

Ref	Lab Sample ID	Sample Point	Date	Time	Test	H2	CO2	O2/Ar	N2	CO	CH4	Hexane	H2S	COS
	•	•			Segment							Plus	(ppmv)	(ppmv)
27	051143-109	G-8	3/29/2005	21:01	2B-TS2								977	81.2
28	051143-110	G-14	3/29/2005	22:00	2B-TS2								54.0	10.3
29	051143-111	G-14	3/29/2005	23:20	2B-TS2								<0.10	0.15
30	051143-115	G-14 G-8	3/30/2005	0:17	2B-152 2B-TS2								~0.10	0.15
31	051143-116	G-14	3/30/2005	0:17	2B-132 2B-TS2								<0.10	0.05
	051143-117	G-14 G-19 Blank		0:47	2B-132 2B-TS2								< 0.10	< 0.050
32			3/30/2005											
33	051143-118	G-19	3/30/2005	0:43	2B-TS2	14.3%	16.7%	0.66%	62.4%	4.32%	1.527%	0.011%	4.63	<0.050
34	051143-121	G-14	3/30/2005	1:53	2B-TS3								<0.10	<0.050
35	051143-122	G-19	3/30/2005	1:46	2B-TS3	14.3%	17.1%	0.66%	62.9%	3.55%	1.474%	0.009%	< 0.050	< 0.050
36	051143-123	G-19	3/30/2005	2:20	2B-TS3								< 0.050	< 0.050
37	051143-124	G-8	3/30/2005	3:16	2B-TS3								923	75.5
38	051143-125	G-19	3/30/2005	3:09	2B-TS3	15.1%	17.6%	0.64%	62.2%	2.90%	1.465%	0.008%	< 0.050	< 0.050
39	051143-126	G-14	3/30/2005	3:12	2B-TS3								<0.10	8.65
40	051143-129	G-19	3/30/2005	3:54	2B-TS3	15.3%	18.0%	0.64%	62.1%	2.61%	1.433%	0.008%	< 0.050	< 0.050
41	051143-130	G-14	3/30/2005	4:12	2B-TS3								<0.10	< 0.050
42	051143-131	G-19	3/30/2005	4:30	2B-TS3		10.00/						< 0.050	< 0.050
43	051143-133	G-19	3/30/2005	5:10	2B-TS3	15.6%	18.0%	0.65%	61.5%	2.86%	1.37%	0.007%	0.23	< 0.050
44	051143-134	G-14	3/30/2005	5:19	2B-TS3								<0.10	0.1
45	051143-135	G-19	3/30/2005	5:42	2B-TS3 2B-TS3								0.03*	< 0.050
46	051143-136	G-8	3/30/2005	6:25									928	75.3
47	051143-137	G-14	3/30/2005	6:20	2B-TS3								<0.10	< 0.050
48	051143-138	G-19	3/30/2005	6:16	2B-TS3	14.8%	17.3%	0.65%	62.3%	3.54%	1.42%	0.009%	< 0.050	< 0.050
49	051143-143	G-14	3/30/2005	7:20	2B-TS3								<0.10	<0.050
50	051143-144	G-19	3/30/2005	7:15	2B-TS3	14.3%	16.5%	0.7%	62.4%	4.7%	1.4%	0.007%	< 0.050	< 0.050
51	051143-145	G-19	3/30/2005	7:53	2B-TS3	13.6%	15.7%	0.7%	63.0%	5.6%	1.4%	0.007%	< 0.050	< 0.050
52	051143-146	G-14	3/30/2005	8:20	2B-TS3	12.40/	15 50/	0.450/	(2.10)	= 010/	1 400/	0.00/0/	<0.10	< 0.050
53 54	051143-147	G-19	3/30/2005	8:32 9:30	2B-TS3	13.4%	15.5%	0.67%	63.1%	5.81%	1.42%	0.006%	< 0.050 <0.10	< 0.050 <0.050
54 55	051143-149 051143-150	G-14 G-19	3/30/2005 3/30/2005	9:30	2B-TS3 2B-TS4	13.4%	15.7%	0.69%	62.2%	6.61%	1.46%	0.0076%	0.10	< 0.050
55 56	051143-150	G-19 G-8	3/30/2005	9:43	2B-154 2B-TS4	13.470	15.770	0.09%	02.270	0.0170	1.40%	0.007076	0.27 918	<0.050 74.1
57	051143-157	G-19	3/30/2005	10:30	2B-154 2B-TS4	13.5%	15.7%	0.69%	62.3%	6.34%	1.42%	0.008%		<0.050
58	051143-158	G-19	3/30/2005	10:30	2B-154 2B-TS4	15.570	12.770	0.0570	02.570	0.0470	1.42/0	0.000 /0	<0.050	<0.050
59	051143-159	G-19	3/30/2005	11:07	2B-TS4								< 0.050	< 0.050
60	051143-160	G-19	3/30/2005	11:35	2B-TS4	12.9%	14.8%	0.64%	64.4%	5.85%	1.39%	0.008%		< 0.050
61	051143-161	G-19	3/30/2005	11:55	2B-TS4								< 0.050	< 0.050
62	051143-162	G-19	3/30/2005	12:15	2B-TS4	12.5%	14.3%	0.64%	65.3%	5.97%	1.28%	0.005%	< 0.050	< 0.050
63	051143-165	G-19	3/30/2005	14:05	2B-TS5	12.9%	14.9%	0.64%	64.7%	5.64%	1.31%	0.006%	< 0.050	< 0.050
64	051143-166	G-8	3/30/2005	14:15	2B-TS5								910	72.9
65	051143-167	G-14	3/30/2005	14:20	2B-TS5								<0.10	0.05
66	051143-176	G-19	3/30/2005	15:13	2B-TS5	12.3%	14.3%	0.64%	65.4%	6.08%	1.29%	0.005%		<0.050
67	051143-177	G-14	3/30/2005	15:20	2B-TS5							0 00 00	<0.10	0.05
68	051143-178	G-19	3/30/2005	16:15	2B-TS5	12.3%	14.4%	0.64%	65.4%	6.04%	1.28%	0.006%		<0.050
69 70	051143-179	G-14	3/30/2005	16:18	2B-TS5	12 49/	14 29/	0 629/	65 89/	E 620/	1 409/	0.0060/	<0.10	0.05
70 71	051143-182 051143-183	G-19 G-14	3/30/2005 3/30/2005	16:55 16:57	2B-TS5 2B-TS5	12.4%	14.2%	0.63%	65.8%	5.63%	1.40%	0.006%	<0.050 <0.10	<0.050 0.01*
71	051143-183 051143-184	G-14 G-14	3/30/2005 3/30/2005	16:57	2B-185 2B-T85								<0.10 <0.10	0.01*
72	051143-185	G-14 G-19	3/30/2005	17:57	2B-185 2B-T85	12.2%	14.2%	0.63%	65.5%	6.09%	1.46%	0.008%		<0.02*
74	051143-204	G-19 G-8	3/30/2005	0:20	2B-135 2B-TS6	10.5%	14.2 %	0.03%	63.9%	11.3%	1.40%	0.003 %		70.8
75	051143-215	G-8	3/31/2005	4:35	2B-130 2B-TS6	10.5%	12.5%	0.75%	63.4%	11.5%	1.37%	0.005%		115
76	051143-234	G-14	3/31/2005	11:35	2B-150 2B-TS7	1011/0	1210 /0	0	00.170	110/0	1.0770	0.00070	<0.10	<0.05
77	051143-235	G-14 G-8	3/31/2005	11:55	2B-TS7	12.7%	12.7%	0.66%	58.0%	13.6%	2.33%	0.019%		53.4
78	051143-236	G-19	3/31/2005	12:53	2B-TS7/8								< 0.050	0.04*

 Table 26 - Summary of Batch Sample Gas Analyses during the NGC-OPT2B Test Campaign

### **11.4.3 FTIR Measurements**

Selected results obtained with the FTIR instrument (installed in the FFTF second floor analytical station) are presented graphically in Figure 62 through Figure 64 for the NGC-OPT2A campaign and in Figure 65 through Figure 67 for the NGC-OPT2B campaign. Throughout these campaigns, the FTIR instrument took samples from any of the three sampling points G-8 (CF-R inlet), G-13 (CF-R outlet), or G-14 (conditioned syngas at the TF-R inlet). At any of these sampling locations the FTIR provided online measurements of the concentrations of a number of gaseous species, including CO<sub>2</sub>, CO, H<sub>2</sub>O, CH<sub>4</sub>, NH<sub>3</sub>, HCl, etc.

Figure 62 shows the measured HCl concentration at G-8, G-13, and G-14 throughout the NGC-OPT2A campaign. The FTIR measured concentrations are generally consistent with the concentrations measured via impingers/ion chromatography (Table 23). Because of char interference with sampling at the SE&C Skid #1, the measured HCl concentrations at the G-13 (~ 11 ppmv on the average) sampling location are higher than those at G-8 (ranging from about 3 to 8 ppmv). The measured HCl concentrations in the conditioned syngas at the G-14 location were well within the 1 to 5 ppmv range, with an average of ~ 2.7 ppmv. The fluctuations shown in the measured HCl concentration at G-13 during the 2A-TS3 testing period were caused by operating difficulties. At about 13:00 on 3/24/05 the FT-IR inlet/outlet heater was found to be off and was turned back on. This explains the preceding HCl concentration fall and subsequent rise (to ~ 30 ppmv) when the heater was back on.

Figure 63 shows the measured NH<sub>3</sub> concentration at G-8, G-13, and G-14 throughout the NGC-OPT2A campaign. The raw syngas derived from the gasification of the washed Indian coal appears to contain about 1000 ppmv NH<sub>3</sub>. During most of the NGC-OPT2A campaign, the measured NH<sub>3</sub> concentration averaged about 1000 ppmv at G-8, 1034 at G-13, and 1117 at G-14. As gasifier performance improved during the 2A-TS5 testing period, the measured NH<sub>3</sub> concentration showed a declining trend. The NH<sub>3</sub> concentration at G-13 averaged about 953 ppmv and that at G-14 about 842 ppmv.

Figure 64 reports the measured H<sub>2</sub>O concentration at G-8, G-13, and G-14 throughout the NGC-OPT2A campaign. The measured CO concentration is also superimposed on this figure. Both species concentrations appear stable during the various measurement periods. The measured H<sub>2</sub>O concentration averaged about 36.6 vol% at G-8, about 37.1 vol% at G-13, and about 34.4 vol% at G-14. The measured CO concentration (in the wet syngas) averaged about 12.6 vol% at G-8, 12.9 vol% at G-13, and 9.8 vol% at G-14. The reduced CO concentration in the conditioned syngas stream at G-14 was due to the watergas shift reaction taking place within the SGB vessel, as explained earlier. These measured values can be converted to a dry syngas basis and compared to results obtained with batch samples to assess the accuracy of the FTIR instrument.

Figure 65 through Figure 67 for the NGC-OPT2B campaign can be interpreted similarly. Compared to the NGC-OPT2A campaign with washed Indian coal, lower HCl concentrations were measured (Figure 65), reflecting the lower chloride content of lignite. However, the HCl concentration in the conditioned syngas was maintained within the desired 1 to 5 ppmv range. The raw syngas (at G-8) steam content averaged about 36.3 vol% during operations with washed Indian coal and about 30.4 vol% during operations with lignite. The measured H<sub>2</sub>O concentrations averaged about 35.3 vol% at G-13 and about 32.2 vol% at G-14, with both series of measurements taken during operations with Indian coal. These differences relate to differences in operating temperature and heat losses associated with the gasifier during operations with Indian coal and lignite. The data shown in Figure 66 indicate that the measured NH<sub>3</sub> concentration during operations with Indian coal were slightly lower than those made during the NGC-OPT2A campaign and that significantly higher NH<sub>3</sub> concentrations (~ 3000 to 3350 ppmv compared to ~ 1000 ppmv) were measured during operations with lignite.

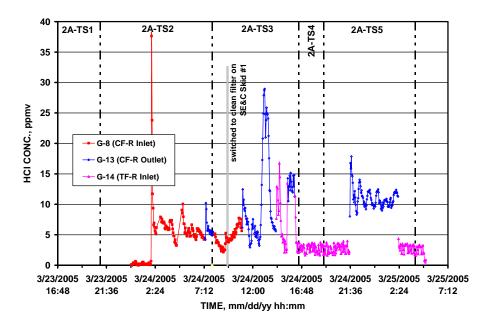


Figure 62 – FTIR Measured HCl Concentration in the NGC Process Section during the NGC-OPT2A Campaign

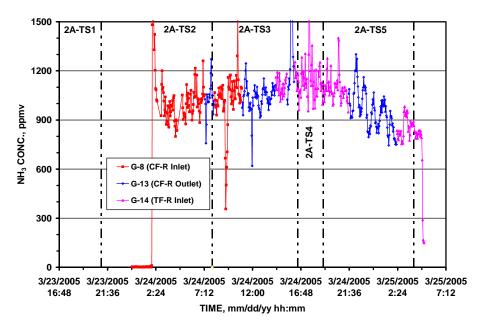


Figure 63 - FTIR Measured NH<sub>3</sub> Concentration in the NGC Process Section during the NGC-OPT2A Campaign

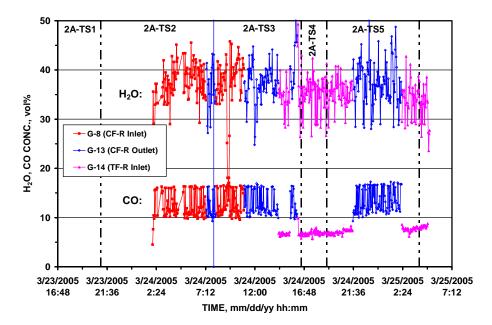


Figure 64 - FTIR Measured H<sub>2</sub>O and CO Concentrations in the NGC Process Section during the NGC-OPT2A Campaign

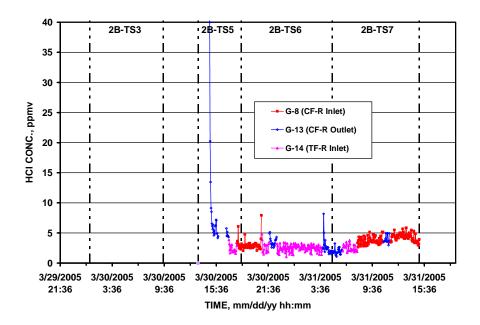


Figure 65 - FTIR Measured HCl Concentration in the NGC Process Section during the NGC-OPT2B Campaign

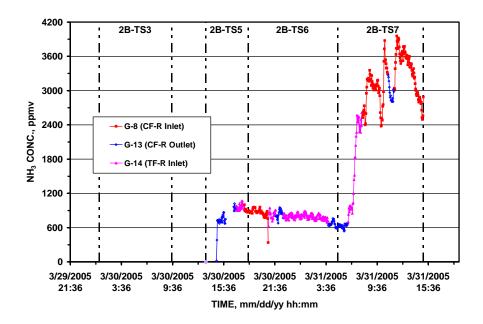


Figure 66 - FTIR Measured NH<sub>3</sub> Concentration in the NGC Process Section during the NGC-OPT2B Campaign

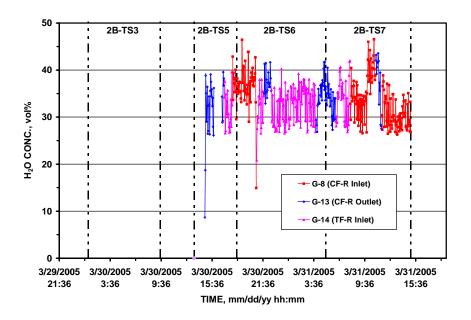


Figure 67 - FTIR Measured H<sub>2</sub>O Concentration in the NGC Process Section during the NGC-OPT2B Campaign

# 12. NOVEL GAS CLEANING OPTIONAL PROGRAM TEST CAMPAIGN #3 (NGC-OPT3)

# 12.1 Objectives

The primary objective of the NGC-OPT3 was to evaluate the performance of the Test Filter-Reactor (TF-R) for mercury removal to very stringent levels (> 90%) from syngas derived from the gasification of a carbonaceous feedstock. The Conditioning Filter-Reactor (CF-R) was to be operated under optimum operating conditions (~ 830°F, 1-10 pulse cleaning frequency, and trona injected at optimum Na-to-Cl ratio when it is necessary to control HCl levels in the CF-R outlet). During selected test periods, Stage II sulfur and halide sorbents would be injected, simultaneously with the Hg sorbent, into the conditioned syngas upstream of the TF-R to assess their effects on the Hg sorbent performance. This was accomplished through a series of test segments to:

(1) set the Flex-Fuel gasifier operating conditions for producing syngas from the test feedstock (North Dakota Lignite) at 250 psig pressure, preferably with a syngas flow rate at the secondary cyclone exit of at least 3,300 lb/hr.

(2) determine the operating conditions for the entire NGC Process "Conditioning" section to consistently result in a syngas with the target characteristics at the TF-R inlet: 1-5 ppmv total sulfur (H<sub>2</sub>S + COS), 1-5 ppmv halide (HCl),  $\sim < 0.1$  ppmw particulate content, a temperature of 400 to 572°F (204 to 300°C), a pressure of  $\sim 225$  psig, and a flow rate equivalent to a face velocity of at least 3 ft/min. The "Conditioning" section includes the Direct Spray Water Quench (DSQ) system for partial cooling of the raw syngas, the CF-R (including pulsing) vessel for particulate control, the Sorbent (trona) Injection system for bulk HCl removal, a Sulfur Guard Bed (and associated pre-heater and slipstream syngas by-pass) for total sulfur control within the desired range at the TF-R inlet, and a Trim Cooler (indirect heat exchanger) for temperature control.

(3) operate the TF-R to confirm the feasibility of removing > 90% of the total mercury compounds (essentially elemental mercury, Hg<sup>0</sup>), via selected dry, fine sorbent injection into the "conditioned" syngas upstream of the TF-R vessel. The key TF-R operating parameters include sorbent-to-mercury mass ratio (i.e., mercury sorbent feed rate), temperature, and pressure (i.e., face velocity).

(4) confirm the effectiveness of "conventional" syngas sampling and measurement via gold traps from slipstreams provided by sample extraction & conditioning (SE&C) systems at sampling locations G-8 (CF-R inlet), G-13 (CF-R outlet), G-14 (conditioned syngas), and G-19 (TF-R product syngas) with total mercury concentrations ranging from ~ 0.5 to 2 ppbv (~ 4.1 to 16.4  $\mu$ g/m<sup>3</sup>) at the TF-R inlet to ~ 0.025 to 0.1 ppbv (0.205 to 0.82  $\mu$ g/m<sup>3</sup>) at the TF-R outlet.

(5) provide representative slipstream samples from the G-19 sampling location (Test Filter-Reactor outlet) for comprehensive, continuous, sensitive, and accurate measurements using a state-of-the-art on-line mercury measurement instrument (Tekran Mercury Vapour Analyzer 2537A).

# 12.2 Process Flow Diagram for NGC-OPT3

For this test campaign in the Flex-Fuel Test Facility (FFTF), the syngas flowed through the entire NGC Process pilot-scale test facility (Figure 6), which includes a raw syngas "Conditioning" section and a "Test" section for deep cleaning of the conditioned syngas stream.

This test involved both Siemens filter-reactors and it was performed similar to the second test campaign (NGC-OPT2A and NGC-OPT2B), except that the Trim Cooler (intermediate indirect heat exchanger) would be operated to further reduce the temperature of the Sulfur Guard Bed outlet syngas to the desired level in the range of ~ 400 to 500°F.

During the initial part of the test campaign, representative samples of the syngas were extracted, conditioned (de-dusted, cooled, and de-pressured), and analyzed with suitable instruments at two main locations:

G-8: Partially-cooled raw syngas (between DSQ and CF-R)

G-13: Partially-conditioned CF-R product syngas (essentially dust-free, but still containing all the raw syngas sulfur and chlorine).

As indicated in Figure 6, the NGC "Conditioning" section also comprises a Sulfur Guard Bed (SGB) and a Trim Cooler (indirect heat exchanger) for bulk sulfur removal and temperature reduction of the CF-R product gas to meet the requirements of the inlet syngas to the "Test" section. Once conditioned, the syngas stream is then introduced into the TF-R vessel for additional deep cleaning via selected dry, fine sorbents that are injected separately into the syngas immediately upstream of the TF-R vessel. Representative samples of the syngas would be extracted, conditioned (de-dusted, cooled, and depressured), and analyzed with suitable instruments at two additional locations:

G-14: Conditioned syngas (TF-R inlet)

G-19: Ultra-cleaned syngas (TF-R outlet)

Other analytical work would also be performed on various samples to characterize gasifier performance and efficiency in generating syngas from the test feedstock, to characterize process emissions for permitting activities, and to assess disposal options for the gasification and syngas cleanup byproduct materials. As shown in Figure 17, process samples include fuel feedstock, ash, dust, raw syngas, fresh sorbent materials, spent sorbents, and condensed liquids, in addition to several gas samples throughout the NGC Process section.

# 12.3 Run Sequence / Chronology

To accomplish test objectives, a sequence was planned similar to the previous test campaigns. This is summarized briefly in Table 27, where the various test segments are defined.

~		
Lignite		
6/7/2005 at ~ 08:00		
6/10/2005 at ~ 06:00		
70		
	TF-R "pre-coating"	
	with Hg Sorbent #2 (3-	
	<b>TS6</b> )	
6/7/2005 at ~ 08:00	Start	6/9/2005 at ~ 16:00
6/8/2005 at ~ 02:00	End	6/9/2005 at ~ 18:00
18	Duration, hr	2
	Hg Sorbent #2 injection	
	( <b>3-TS7</b> )	
6/8/2005 at ~ 02:00	Start	6/9/2005 at ~ 18:00
6/8/2005 at ~ 14:00	End	6/10/2005 at ~ 04:00
12	Duration, hr	10
	Pulse TF-R	6/10/2005 at ~ 05:00
	Terminate NGC-OPT3	6/10/2005 at ~ 06:00
12		
6/9/2005 at ~ 02:00		
6/9/2005 at ~ 04:00		
2		
	_	
	-	
6/9/2005 at ~ 04:00	_	
6/9/2005 at ~ 04:00 6/9/2005 at ~ 14:00	-	
	-	
6/9/2005 at ~ 14:00	-	
6/9/2005 at ~ 14:00		
	6/10/2005 at ~ 06:00 70 6/7/2005 at ~ 08:00 6/8/2005 at ~ 02:00 18 6/8/2005 at ~ 02:00 6/8/2005 at ~ 14:00 12 6/8/2005 at ~ 14:00 6/9/2005 at ~ 02:00 12 6/9/2005 at ~ 02:00 6/9/2005 at ~ 02:00 6/9/2005 at ~ 02:00	$6/7/2005 \text{ at} \sim 08:00$ $6/10/2005 \text{ at} \sim 06:00$ $70$ TF-R "pre-coating" with Hg Sorbent #2 (3- TS6) $6/7/2005 \text{ at} \sim 08:00$ Start $6/8/2005 \text{ at} \sim 02:00$ End         18       Duration, hr         Hg Sorbent #2 injection (3-TS7) $6/8/2005 \text{ at} \sim 02:00$ Start $6/8/2005 \text{ at} \sim 14:00$ End         12       Duration, hr         Pulse TF-R $6/8/2005 \text{ at} \sim 02:00$ $70$ Terminate NGC-OPT3         12 $70$

 Table 27 - Test Segments Planned in the NGC-OPT3 Test Campaign

The analytical work scope during each test segment planned is highlighted below:

# 3-TS1

- Calibrate CAT 200 Rosemount analyzer
- Complete revamping of G-8, G-13, G-14, and G-19 sampling lines
- Start setting up Hg gold traps, on-line Hg analyzer, and steam impingers
- Clean Sample Extraction & Conditioning Skid #1 filters A and B
- Assemble G-8 gas sampling cylinders (after cleaning and evacuation by GTI CRS dept.)
- Prepare data sheets, tags, labels, etc.

# 3-TS2

- Set up impingers for water measurements at G-8 only (these are needed to estimate raw syngas H<sub>2</sub>O content).
- Complete setting up gold traps for Hg sampling for subsequent measurement off-line using a Nippon WA-4 Mercury Analyzer.
- Complete setting up Tekran Mercury Vapour Analyzer 2537A instrument for on-line analysis of Hg.
- When gasifier is operating at close to 250 psig:
  - Make 3 Hg measurements at G-8 (these will provide an indication of the Hg level released in the syngas. Note: char may be affecting these measurements.
  - How much Hg should we expect to measure?

# $[Hg^{0}]_{o} (ppbv) = 1000 * [MW_{sg} * F * M] / [Qr * MW_{Hg}]$

- $[Hg^0]_0$  = Measured total mercury (Hg<sup>0</sup>) concentration in raw (wet) syngas (at G-8), ppbv
- Q<sub>r</sub> = Raw (wet) syngas mass flow rate, lbs/hr
- MW<sub>sg</sub> = Molecular weight of raw syngas, lbs/lb-mole
- F = Coal feed rate to gasifier, lbs/hr
- M= Coal mercury content, ppmw

<u>Example:</u> A coal containing about 0.06 ppmw Hg (e.g., processed Saskatchewan lignite) fed to the Flex-Fuel gasifier at a rate of 650 lbs/hr to generate a raw, wet syngas stream of 3,000 lbs/hr with an average MW of 25, the estimated syngas mercury content is ~ 1.62 ppbv, ~ 13.29  $\mu$ g/m<sup>3</sup> (1 ppbv = 8.2041  $\mu$ g/m<sup>3</sup>). This is well above the detection limit of the Nippon WA-4 Mercury Analyzer (0.01  $\mu$ g/m<sup>3</sup>).

- Make 3 Hg measurements at G-13. At this sampling location syngas is essentially dustfree and we should expect that these will be the highest levels we'll measure beyond the CF-R.
- Solids analysis: to estimate carbon conversion and Hg balance (lignite feed, gasifier ash, secondary cyclone fines, SE&C Skid #1 char).
- Gas sampling at SE&C Skid #1: take 3 samples towards the end of 3-TS2 for mass balance considerations. Note: no gas sampling is required at the other skids (beyond G-8).

# 3-TS3

- Test Filter-Reactor (TF-R) at  $T_1 \approx 350^{\circ}$ F (above steam condensation point? Verify)
- Send entire syngas stream through Sulfur Guard Bed (SGB) (Sorbent 1 does not require a particular sulfur level in the syngas to work effectively since it had already been "especially

sulfided" by supplier (Synetix/Johnson Matthey). Note: we will not have the HP 5890 GC set up at G-14 to measure  $H_2S$  (and COS) level in the conditioned syngas. SGB breakthrough an issue?

- Hg sampling: up to 8 samples at G-13; up to 6 samples at G-14; and up to 3 samples at G-19. G-14 Hg samples are needed to assess effect of SGB on Hg concentration in the syngas. G-19 samples will determine effect of "empty" TF-R on Hg in the syngas (ideally, G-14 and G-19 samples should be the same)
- G-8: up to 6 (one every 2 hours) gas samples at G-8 (select which to submit for analysis in consultation with FFTF operating crew).
- Water impingers at G-8: up to 6
- Solids analysis: T-502 (gasifier ash), T-402 (secondary cyclone fines): 2 samples every hour. Determine which samples should be analyzed (this work can be done post-test depending on budget and need)

# 3-TS4 and 3-TS5

Hg Sorbent Injection Rate (grams/min) =  $[453.6/(60 * 10^{6})] * F * M * MR$ 

- F = Coal feed rate to gasifier, lbs/hr
- M= Coal mercury content, ppmw
- MR= Sorbent-to-mercury mass ratio

Example: A coal containing about 0.06 ppmw Hg (e.g., processed Saskatchewan lignite) fed to the Flex-Fuel gasifier at a rate of 650 lbs/hr to generate a raw, wet syngas stream of 3,000 lbs/hr with an average MW of 25, the estimated syngas mercury content is ~ 1.62 ppbv, ~ 13.29  $\mu$ g/m<sup>3</sup> (1 ppbv = 8.2041  $\mu$ g/m<sup>3</sup>). This is equivalent to ~ 13 ppbw Hg. For a Hg sorbent-to-mercury mass ratio (MR) of 2000, the Hg sorbent feed rate into the conditioned syngas upstream of the TF-R is ~ 0.59 gram/min.

- Hg sampling: up to 8 samples at G-14; up to 6 samples at G-19; and up to 3 samples at G-13 (if possible). G-14 Hg samples are needed to monitor variations in syngas Hg content, inlet to the TF-R. G-19 samples will determine extent of Hg removal via dry sorbent injection in the TF-R. G-13 samples may be a good idea to see effect of SGB as more sulfur is loaded onto the Süd-Chemie SGB material.
- G-8: up to 6 (one sample every 2 hours) gas samples at G-8 (select which to submit for analysis in consultation with FFTF operating crew).
- Water impingers at G-8: up to 6
- Solids analysis: T-502 (gasifier ash), T-402 (secondary cyclone fines): 2 samples every hour. Determine which samples should be analyzed (this work can be done post-test depending on budget and need)

### 3-TS6 and 3-TS7

- Hg sampling: up to 8 samples at G-14; up to 6 samples at G-19; and up to 3 samples at G-13 (if possible). G-14 Hg samples are needed to monitor variations in syngas Hg content, inlet to the TF-R. G-19 samples will determine extent of Hg removal via dry sorbent injection in the TF-R. G-13 samples may provide insight into the effect of SGB as more S is loaded onto the Süd-Chemie SGB material.
- G-8: up to 6 (one every 2 hours) gas samples at G-8 (select which to submit for analysis in consultation with FFTF operating crew).
- Water impingers at G-8: up to 6
- Solids analysis: T-502 (gasifier ash), T-402 (secondary cyclone fines): 2 samples every hour. Select samples for analysis).

#### Post-test analytical work

- Char from SE&C Skid #1
- CF-R char
- TF-R spent sorbent/char mix
- Etc.

#### 12.4 Results and Discussion - NGC-OPT3

The NGC-OPT3 test campaign was initiated on Wednesday June 8, 2005 with lignite (Fort Union, ND). Unfortunately, several feed interruptions were experienced, which were caused by plugging of the gasifier fluid-bed discharge. This part of the test, designated as NGC-OPT3A, had to be aborted the following day, on Thursday June 9, 2005. A plugged feed screw equalization line was suspected, confirmed, and cleared. The piping was modified to eliminate this problem in the future. The NGC Process section was inspected and the Conditioning Filter-Reactor (CF-R) hopper drained for a clean start. The Sulfur Guard Bed (SGB) and Test Filter-Reactor (TF-R) were maintained under a hot nitrogen purge to keep them hot and dry.

Testing was resumed on Sunday June 12 (i.e., NGC-OPT3B) also with lignite. The Flex-Fuel Test Facility (including the NGC Process section) was operated continuously for 51 hours, accomplishing a significant portion of the third test campaign objectives. The major accomplishments and findings from this work included:

- GTI demonstrated the capability to measure mercury at parts-per-billion levels in coal-derived syngas and obtain reasonable material balance.
- There was evidence of a significant level of mercury capture (50-75%) with the highertemperature Hg removal sorbent (TDA sorbent) at the relatively high temperature of 572°F (300°C, optimum NGC Process Stage II operating temperature).
- Approximately 200 gas, solid, and liquid samples were taken and analyzed, and additional post-test analyses were performed.

### 12.4.1 Gasifier and Filter-Reactor Operations

The Flex-Fuel Test Facility (gasification and Novel Gas Cleaning Process sections) was successfully operated throughout most of the NGC-OPT3B test campaign, including two Siemens' barrier filters (Conditioning Filter-Reactor, CF-R, and Test Filter-Reactor, TF-R), Sulfur Guard Bed, SGB (and associated low-pressure nitrogen pre-heater and slipstream by-pass system), and Stage II sulfur (G-72E) and halide (G-92C) sorbent feeders. The T-2107 Stage II Sulfur Sorbent Feeder was dedicated to feeding the finely-ground mercury sorbents. The T-2108 Stage II Halide Sorbent Feeder was used to feed a

mixture of finely-ground G-72E sulfur sorbent and finely-ground G-92C halide sorbent (in combination with nahcolite to improve its feeding) during the 3B-TS7 test segment when the effect of the Stage II sulfur and halide sorbents on the Hg sorbent mercury removal performance was evaluated. In addition, because only the higher-temperature Hg removal sorbent was evaluated during this campaign, there was no need to engage the Trim Cooler (located between the SGB and TF-R) to reduce the conditioned syngas temperature at the TF-R inlet to  $\sim 400^{\circ}$ F, as had been planned.

Because the raw syngas stream contained steam, it was necessary to heat up the SGB catalyst bed (Süd-Chemie's G-72E catalyst) in the NGC Process "Conditioning" section above the syngas dew point to avoid water condensation, which otherwise would severely damage the catalyst's desulfurization performance. Accordingly, during the NGC-OPT3B campaign the raw syngas was initially routed from the DSQ system (HE-2001-A) through the CF-R vessel (T-2153), and then bypassed directly to the PCV-602 pressure let-down system. Simultaneously, low-pressure nitrogen was fed through the Nitrogen Pre-Heater (SH-2002) into the SGB vessel (R-2002), which had been isolated from the front-end of the NGC Process section (Figure 2 and Figure 31). The hot nitrogen exiting the SGB vessel was then routed around the Trim Cooler (HE-2071) through the Test Filter-Reactor (T-2147), and then through the PCV-2181 pressure let-down system. This made it possible to also pre-heat the TF-R vessel before directing the conditioned syngas stream into it.

Once the "Conditioning" section reached steady state and the TF-R brought to a sufficiently high temperature, the partially-cooled and essentially particulate-free syngas stream exiting the CF-R was sent through the SGB vessel, then bypassed through the PCV-2006, around the Trim Cooler, to the Condensate Knock-Out Tank (T-2072), and then to the TF-R. Pressure in the "Conditioning" section was controlled by the PCV-2003 pressure let-down system, and pressure in the "Test" section was controlled by the PCV-2181 pressure let-down system. In order to evaluate the performance of the highertemperature Hg removal sorbent under optimum Stage II operating conditions for desulfurization and dechlorination sorbents in the "Test" section of the NGC Process, the temperature in the TF-R should be in the 550°F to 575°F range. Since no trona injection was necessary in the "Conditioning" section (lignite contained even less chlorine than the washed Indian coal), the operating temperature for the CF-R vessel was determined primarily by the requirements of the SGB vessel. Therefore, the temperature strategy adopted involved setting the inlet temperature to the CF-R vessel so that, with ambient heat losses from the intervening vessels and piping, the operating temperature of the TF-R vessel would be in the optimal range. Moreover, to minimize mercury/char interactions in the CF-R vessel, the inlet temperature was maintained slightly higher than in previous tests by slightly modifying the operating conditions of the DSO system. This also helped ensure the inlet temperature to the SGB was sufficiently high to prevent early breakthrough of the SGB (the same, partially utilized catalyst load from previous tests was used).

During the third test campaign, raw syngas, derived from the gasification of North Dakota lignite (0.89% S, ~ 122 µg/g Cl, 0.06 µg/g Hg), was successfully conditioned to the temperatures and contaminant levels required at the inlet to the TF-R. Throughout testing, temperatures across the NGC Process section were maintained at ~ 1450°F at the secondary cyclone, ~ 1400°F at the inlet to the Direct Spray Water Quench, ~ 700°F at the inlet to the CF-R, ~ 650°F at the outlet of the CF-R, ~ 650°F at the SGB inlet, ~ 600°F at the SGB outlet, ~ 600°F at the TF-R inlet, and ~ 550°F at the TF-R outlet. These conditions ensured that the SGB was operated at optimal conditions for maximized utilization of the SGB catalyst and efficient sulfur removal. These conditions also ensured that contaminant removal reactions across the TF-R vessel were kept within the optimum temperature range (550°F to 575°F). The total sulfur concentration (H<sub>2</sub>S and COS) at the inlet was estimated to be within the 1-5 ppmv range throughout testing, although the entire syngas stream was passed through the SGB vessel (the on-line GC/FPD that provided near-continuous H<sub>2</sub>S and COS measurements during the first and second test campaigns was not available for this third test campaign). As indicated above, there was no need to engage the Trim Cooler for this test campaign. In addition, because of low levels of HCl in the raw syngas (~ 20 ppmv) and some measured HCl removal in the CF-R vessel (through interaction with char) and in the SGB vessel (a

small amount of CaO in the SGB catalyst), it was not necessary to engage the trona sorbent feeder upstream of the CF-R for bulk HCl removal. No impinger sampling was performed for HCl measurement via ion chromatography in this campaign and the on-line FT-IR instrument was not available for HCl analysis. However, based on the NGC-OPT2A and NGC-OPT2B test campaign results, the conditioned syngas HCl content was probably ~ 1 ppmv or lower. Results from this test campaign are discussed in more detail below.

Conditioning the raw syngas (at G-14, TF-R inlet), performing Hg measurements in the syngas throughout the NGC Process section, and evaluating the mercury removal performance of the higher-temperature Hg sorbent ("activated" and finely-ground TDA sorbent) were the focus of this third test campaign (NGC-OPT3B). This campaign comprised the test segments shown in Table 28. As indicated, gasification of metallurgical coke (Bethlehem Coke Breeze, for second-stage heating up of the gasifier) and then North Dakota lignite occurred over ~ 51 hours, from ~ 01:00 on 6/13/2005 through 04:00 on 6/15/2005. The period on lignite coal, during which mercury measurements and removal testing were performed, began at ~ 21:00 on 6/13/2005 and continued for 36 hours, until the test was terminated. The gasification section was operated under steady state conditions throughout most of these test segments as demonstrated by the stable gasifier output shown in Figure 68. In this figure the hydrogen (H<sub>2</sub>), carbon monoxide (CO), and methane (CH<sub>4</sub>) concentrations in the raw, dry product syngas are given, as measured by the Rosemount CAT 200 online analyzer at the G-8 sampling location (CF-R inlet). The noticeable changes in the CO concentration during the 3B-TS6 test segment, from ~ 21:45 on 6/14/05 to ~ 02:00 on 6/15/05 are related to changes in the gasifier operating conditions that were implemented to circumvent some difficulties experienced with the DSQ system.

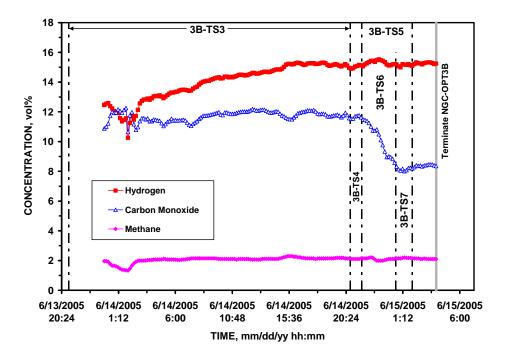


Figure 68 - H<sub>2</sub>, CO, and CH<sub>4</sub> in the Raw Gasifier Product Syngas at G-8 (CF-R Inlet) during the NGC-OPT3B Test Campaign

 Table 28 - Test Segments in the NGC-OPT3B Test Campaign (June 13-15, 2005)

	NGC-OPT3B

Overall	
Start	6/13/2005 at ~ 01:00
End	6/15/2005 at ~ 04:00
Duration, hr	51
System heat up with Start-Up Heater & second-stage gasifier heating with met coke (3B-TS1)	
Start	6/13/2005 at ~ 01:00
End	6/13/2005 at ~ 16:00
Duration, hr	15
Establishing Steady State with Lignite coal (3B-TS2)	
Start	6/13/2005 at ~ 16:00
End	$\frac{6}{13/2005}$ at $\sim 21:00$
Duration, hr	5
Establishing Preferred Conditions in NGC Process Section (3B-TS3)	
Start	6/13/2005 at ~ 21:00
End	6/14/2005 at ~ 20:45
Duration, hr	~ 24
TF-R "pre-coating" with Hg "higher-temperature" sorbent (3B-TS4)	
Start	6/14/2005 at ~ 20:45
End	6/14/2005 at ~ 21:45
Duration, hr	1
Hg "higher-temperature" sorbent injection (3B-TS5)	
Start	6/14/2005 at ~ 21:45
End	6/15/2005 at ~ 02:00
Duration, hr	~ 4
Hg "higher-temperature" sorbent injection (3B-TS7)	
simultaneously with S & halide Stage II sorbent mix	6/15/2005 at 00.25
Start End	6/15/2005 at ~ 00:35 6/15/2005 at ~ 02:00
Duration, hr	<u>- 6/15/2005 at ~ 02:00</u> 1.5
	1.3
Terminate NGC-OPT3	6/15/2005 at ~ 04:00
Dulas TE D	Doct tost
Pulse TF-R	Post-test

#### **12.4.1.1** Gasifier Performance

#### <u>Summary</u>

Throughout this report, gasification of the PDU test program carbonaceous feedstocks (Bethlehem Coke Breeze, washed Indian coal, and North Dakota lignite) is reported only in the context of the Novel Gas Cleaning Program testing. Given that the lignite used is a domestic fuel, its gasification is discussed in more detail in the section below (similar data are provided in Appendix B for operation with washed Indian coal during the second test campaign).

As mentioned earlier, GTI arranged with the Green River Energy Coal Creek Station (GRE/CCS) to have about 100 tons of Fort Union lignite processed (crushed, screened, and dried) to meet the Flex-Fuel gasifier specifications. GRE's fluidized-bed drying process produced three streams: product, elutriates, and undercuts, with a surface moisture of ~ 0% and inherent moisture of ~ 20%. To preserve the quality of the lignite and more importantly the contaminant levels for the PDU test program (especially mercury content), the product and elutriates fractions were blended and the undercuts recrushed, screened, and stored in proper containers for additional blending as necessary. Samples from the lignite fed throughout the third test campaign indicated the lignite feed contained an average of ~ 0.084 ppmw of mercury, compared to the 0.093 ppmw estimate provided by GRE (Table 15). It should also be noted that the lignite used contained ~ 5.4% Na<sub>2</sub>O.

Lignite was first gasified during the latter stages of the second test campaign (NGC-OPT2B), where its suitability was confirmed as the feedstock of choice for the third test campaign. The results obtained in this testing demonstrated the technical feasibility of the filter-reactor concept and the ability of the NGC Process to deep clean the gasifier product gas to very stringent levels (10-50 ppbv for H<sub>2</sub>S, COS, and HCl). The same cleaning efficiencies were measured with lignite as were achieved with the washed Indian coal, showing that the NGC Process functioned very well with two diverse fuels.

This third test campaign, which focused on the evaluation of mercury capture with sorbents at relatively high temperatures, used lignite exclusively as the source of syngas and provided another opportunity to demonstrate efficient gasification of lignite in the fluidized bed gasifier. Hot operation extended for a total of 48 hours, gasifying approximately 36,000 lbs (18 tons) of lignite. The Conditioning Filter-Reactor (CF-R) was online through all startup, steady-state, and shutdown operations (100% availability). The maximum lignite coal feed rate was ~ 750 lbs/h and the longest continuous operating period was 36 hours, yielding balances at 250 psig operation.

Steady-state air-blown operation at true ash-balanced conditions was achieved at 250 psig during this third test campaign, with excellent material and energy balances (Table 29). MAF (moisture- and ash-free) coal and carbon conversions have been estimated at 94% and 91%, respectively (Table 30). Table 31 provides particle size distributions for samples from the lignite gasifier feed, classifier discharge, secondary cyclone fines, and Conditioning Filter-Reactor fines. The dry product gas consisted of approximately 14% H<sub>2</sub>, 14% CO<sub>2</sub>, 13% CO, 2% CH<sub>4</sub>, 57% N<sub>2</sub>, and a total sulfur (H<sub>2</sub>S + COS) concentration of ~ 1,150 ppmv (Table 35). The corresponding nitrogen purge free heating value of the dry syngas was 123 Btu/scf (Table 30).

Based on the above performance a syngas heating value of over 150 Btu/scf can be expected in a commercial-scale gasifier with its lower heat losses and more efficiently integrated plant design. Carbon conversion in the 95-98% range should also be achievable. The GTI U-GAS® gasification process is therefore quite suitable for scale-up to commercial power generation applications. Indeed, this was the subject of a recent study by GTI and Nexant on lignite-based IGCC using the GTI U-GAS® gasification technology together with currently available technologies for coal crushing and drying, coal feeding, and gas cleaning. Sensitivity of the economics to various technical and economic factors was also explored in this study. In addition to the feasibility of scaling up GTI's gasification technology for power generation

with lignite, this study showed that incremental improvement in coal and gas handling systems and overall plant integration could further improve the economics of this approach.<sup>14</sup>

#### Gasifier Performance Determination Procedures

Several assumptions/adjustments/simplifications are typically made in the assessment of gasifier performance. The assumptions made in the lignite gasification case are listed below:

- 1. The ultimate analysis of lignite coal feed (Table 14) was adjusted to make the ash content consistent with the proximate analysis.
- 2. The syngas analyses (Table 35) were adjusted to an oxygen free basis. The moisture content was assumed to be  $\sim 25$  vol% based on impinger sampling data.
- 3. The Conditioning Filter-Reactor solids discharge rate was determined based on the actual fines collected from the filter vessel divided by the test duration (780.4 lbs during 36 hours, or about 21.7 lb/hr). This is very close to 3% of the coal feed rate that is typically assumed in this type of estimations.
- 4. The heat loss was calculated based on an estimated total surface area for the system (gasifier, cyclones and diplegs, cooler, filter, and connecting piping), 200°F shell temperature, 60°F ambient temperature, 2.42 Btu/ft<sup>2</sup>-h combined radiation and convection heat transfer coefficient, zero wind speed, and 0.96 surface emissivity.
- 5. The measured heating values of the classifier, cyclone, and filter solids were used for the heat balance.

Steady state was selected to correspond to the ash-balanced operating period from 09:00 to 19:00 on 6/14/05, by examining the quick ash data (Table 36 and Table 34). During this steady-state period, coal conversion was estimated at 94% and the gas heating value at 123 Btu/scf.

Flex Fuel Test: Lignite Coal Period: From: 6-14-05 09:00 To: 6-14-05 19:00	COA PURGE/PULSI CONVEY N2	~		IER		AIR		FILTER FILTER DISCHARGE	🄶 SYNGA	s
					I FLEX FUEL					
Stream No Stream Discription Temperature, °F Solids Flow, Ib/h Dry Solids Composition wt%	1 Coal Feed 60 709	<b>2</b> Air Feed 112	3 Steam Feed 439	4 Nitrogen Feed 90	5 Classifier Discharge 952 68	6 Cyclone Discharge 1318 20	7 Water Feed 60	8 Filter Discharge 706 22	9 Product Syngas 706	
Dry Solids Composition, wt% Ash Carbon Hydrogen Nitrogen Sulfur Oxygen Total Liquid Flow, Ib/h	14.18 59.30 3.86 0.90 0.87 <u>20.89</u> 100.00				70.93 27.07 0.33 0.33 0.80 <u>0.54</u> 100.00	69.67 27.40 0.32 0.26 2.35 <u>0.00</u> 100.00	285	58.62 35.50 0.42 0.34 1.50 <u>3.62</u> 100.00		
Gas Flow, lb/h		1527	195	209			280		2936	
Gas Composition, mol% H2 CO2 O2 N2 CO CH4 C2H6 C2H4 C2H4 C6+ H2S COS		21.00 79.00		100.00					10.57 10.30 0.00 42.53 9.67 1.82 0.01 0.00 0.01 0.00 0.01	
H2O Total		<u>0.00</u> 100.00	<u>100.00</u> 100.00	<u>0.00</u> 100.00					<u>25.00</u> 100.00	
10(0)		100.00	100.00	100.00					100.00	
INPUT STREAMS	Carbon	Hydrogen	Oxyge	en	Nitrogen	Sulfur	Ast	<u> </u>	otal	Btu/h
Coal Feed Air Feed Steam Feed N2 Feed	342.3	37.1 21.8	237 355 173	.6 .0	5.2 1171.3 208.9	5.0	8		709.3 1526.9 194.8 208.9	5.89E+06 1.80E+04 2.40E+05 1.50E+03
Water Feed Total Input	342.3	31.9 90.8	253 1019		1385.4	5.0	8	1.9 2	285.3 2925.3	0.00E+00 6.15E+06
OUTPUT STREAMS										
Product Syngas Classifier Discharge 2nd Cyclone Discharge Filter Discharge Heat Loss	319.0 18.4 5.5 7.7	96.3 0.2 0.1 0.1	0 0	.4 .0 .0	1445.2 0.2 0.1 0.1	3.3 0.5 0.5 0.3	14 13	9.4 4.0 3.5	2936.2 69.1 20.1 21.7	5.06E+06 2.87E+05 8.99E+04 1.14E+05 5.45E+05
Total Output	350.5	96.7	1072		1445.5	4.6			3047.1	6.10E+06
Out - In % Balance (Out/In)	8.2 102.4	5.9 106.5	52 105		60.1 104.3	-0.4 92.6		5.0 3.9	121.8 104.2	-4.71E+04 99.2

# Table 29 – Material and Energy Balance During Steady State Operation with Lignite

(All units in lb/h unless otherwise noted)

GASIFIER OPERATING & PERFORMANCE DATA	
Gasifier Pressure, psig	261
Gasifier Bed Temperature, °F	1539
Coal Feed Rate, lb/h	709
Air Feed Rate, lb/h	1527
Steam Feed Rate, lb/h	195
Steam/Carbon Ratio, lb/lb	0.57
Oxygen/Carbon Ratio, lb/lb	1.04
Gasifier Bed Density, lb/cu ft	29.6
Gasifier Bed Height, ft	8.7
Gasifier Superficial Velocity, ft/s	2.1
MAF Coal Gasification Intensity, lb/cu ft -hr	57
Dry & Purge N2 Free Syngas HHV, Btu/SCF	123
MAF Coal Conversion, %	94

Table 30 – Gasifier Operating and Performance Parameters with Washed Indian Coal

Table 31 – Feed and Discharge Solids Particle Size Distributions

Retained on	Coal	Classifier	Cyclone	Filter
US Sieve, wt %	Feed	Disch	Disch	Disch
6	9	6.0	0.0	0.6
12	23.8	21.8	0.0	0.4
20	26.3	16.8	0.0	0.4
40	21.5	11.8	0.0	1.0
60	11.6	10.3	0.2	1.0
80	4.5	12.0	0.2	1.0
100	1.3	8.1	0.4	0.6
140	1.0	8.1	1.9	2.3
200	0.4	2.4	5.1	2.7
230	0.1	0.6	3.4	1.7
270	0.1	0.3	6.3	2.3
325	0.1	0.2	5.5	2.9
Pan	0.3	1.6	77.0	83.1
Total	100.0	100.0	100.0	100.0

#### **12.4.1.2** Filter-Reactor Operations

Temperature drop and pressure drop behaviors, and key process parameters (inlet syngas temperature, syngas mass flowrate, and operating face velocity) for the CF-R and TF-R vessels are reported in Figure 69 through Figure 72 for the NGC-OPT3B campaign. The figures shown cover the entire test duration, and on each figure the various test segments are delineated consistent with the information provided in Table 28. The Flex-Fuel gasifier pressure was brought up to ~ 250 psig after initiating the gasification of lignite coal (during test segment 3B-TS2). The inlet temperature for the syngas to the CF-R was also reduced from ~ 790°F to ~ 690°F and then raised back to ~ 800-825°F during

the latter stages of the 3B-TS3 test segment. It steadily decreased and then stabilized at ~  $660^{\circ}$ F during the sorbent injection test segments 3B-TS4 and 3B-TS5. Figure 69 shows there were two distinct periods during which process conditions were relatively constant for the CF-R vessel: from ~ 02:30 to 20:45 on 6/14/05 and from 20:45 on 6/14/05 to 04:00 on 6/15/05, when the NGC-OPT3B campaign was terminated. Although in both periods lignite was being gasified, these differences in the CF-R pulsing cycles were caused by changes in the gasifier operating conditions.

The syngas mass flowrate at the CF-R inlet was initially at ~ 2,600 lbs/hr, which corresponds to approximately 2.5 ft/min operating face velocity. Both the syngas flowrate and the face velocity for the CF-R vessel increased during the 3B-TS3 test segment, as shown in Figure 69, as gasifier performance improved. During the steady-state operating period from ~ 09:00 to ~ 19:00 on 6/14/05, the syngas mass flowrate and the CF-R face velocity averaged about 2,850 lbs/hr and 2.77 ft/min, respectively. The solids loading in the syngas was moderate, and the time between pulses varied from ~ 9 to 11 minutes during the first period and 20 to 22 minutes during the second period. As shown in Figure 69, the differential pressure (PDI-2153) was allowed to increase to about 72 in wg before pulsing was initiated. After pulsing the differential pressure was about 38 in wg during the first stable operating period for the CF-R, and about 22 in wg during the second period. Pulsing was performed successfully with the fast-acting valves. The syngas temperature drop across the CF-R vessel appears to be in the range of 40 to 50°F, especially during steady state operating periods.

At ~ 19:15 on 6/14/05, it became necessary for the operating crew to start reducing gasifier pressure to circumvent a rising pressure drop across the DSQ system and continue the test campaign. As the gasifier pressure was reduced, the syngas mass flowrate started to decline, as shown in Figure 71, reaching ~ 2,670 lbs/hr at 20:45 when the 3B-TS4 test segment was initiated. [Note: it was decided to proceed with sorbent injection in the 3B-TS4 test segment despite the occurring changes]. The syngas mass flowrate continued to drop, reaching ~ 2,645 lbs/hr at the end of the 3B-TS4 test segment and ~ 1,885 lbs/hr by ~ 00:35 on 6/15/05 when the 3B-TS7 test segment was started (i.e., "higher-temperature" Hg sorbent injection simultaneously with sulfur and halide Stage II sorbent mix). As shown in Figure 71, throughout the 3B-TS7 test segment and until the campaign was terminated, operating conditions were stable. The syngas mass flowrate averaged about 1,940 lbs/hr during the 3B-TS7 test segment and the face velocity about 2.19 ft/min.

Figure 70 shows the measured temperature and pressure drop behavior for the TF-R vessel. Figure 72 shows the measured inlet syngas temperature and mass flowrate, and the calculated operating face velocity for the TF-R vessel. The syngas temperature at the TF-R inlet was about 650°F at 20:45 on 6/14/05 when the 3B-TS4 test segment was initiated. It showed a decreasing trend and reached about 516°F at the beginning of test segment 3B-TS7. It remained constant at about this level throughout the 3B-TS7 test segment. Similarly, the TF-R outlet temperature decreased from ~ 601°F at 20:45 on 6/14/05 (i.e., start of 3B-TS4) to ~ 478°F at 00:35 on 6/15/05 (start of 3B-TS7). During this latter test segment, the syngas temperature at the TF-R outlet averaged about 455°F. Therefore, it appears that during the 3B-TS7 test segment, the TF-R was operated at a temperature of about 486°F, i.e., ~ 65°F lower than in the second test campaign (NGC-OPT2A and 2B). The syngas mass flowrate at the TF-R inlet and the face velocity profiles showed similar trends to their CF-R counterparts, although increases in the TF-R face velocity towards the latter stages of the 3B-TS3 test segment were more noticeable (Figure 72).

The pressure drop behavior depicted in Figure 70 for the TF-R vessel is distinctly different from the pressure drop behavior for the CF-R vessel (Figure 69). In addition, because of the changes in operating conditions that were implemented to contain the difficulties experienced with the DSQ system, the TF-R pressure drop profile requires close examination. Figure 70 shows that the TF-R  $\Delta P$  ranged between 17 and 20 in wg during most of the 3B-TS3 test segment, and increased to about 22 in wg during the latter stages of this test segment as syngas mass flowrate increased. Because the syngas mass flowrate continued to drop during the 3B-TS4 and the 3B-TS6 test segments, no increases in the TF-R  $\Delta P$  were measured despite the fact that sorbent injection was performed during these test periods. As shown in

Figure 70,  $\Delta P$  across the TF-R continued to drop and reached about 12 in wg. However, during sorbent injection in the 3B-TS7 test segment, which, as pointed out above, was performed under stable operating conditions, the TF-R  $\Delta P$  is seen to increase, evidence that at least portions of the sorbents being injected were reaching the filter elements (candles).

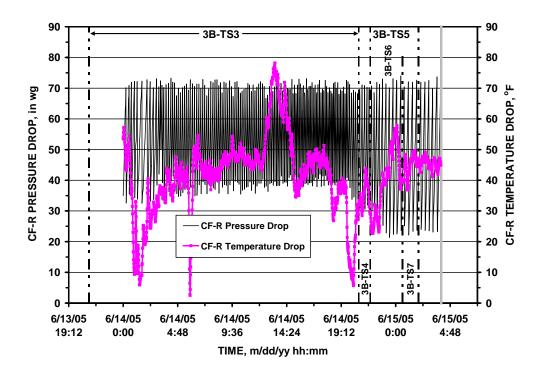


Figure 69 - Temperature and Pressure Drop Behavior of the Conditioning Filter-Reactor during the NGC-OPT3B Campaign

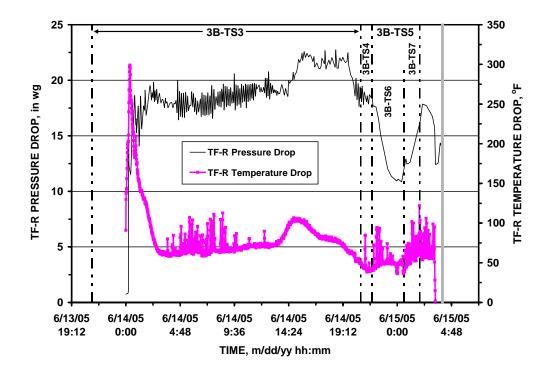


Figure 70 - Temperature and Pressure Drop Behavior of the Test Filter-Reactor during the NGC-OPT3B Campaign

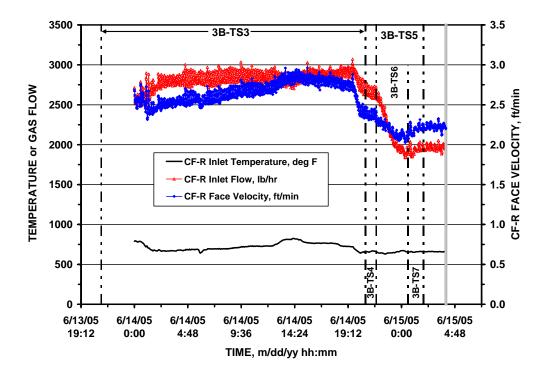


Figure 71 - Temperature, Syngas Flowrate, and Face Velocity at the Conditioning Filter-Reactor Inlet during the NGC-OPT3B Campaign

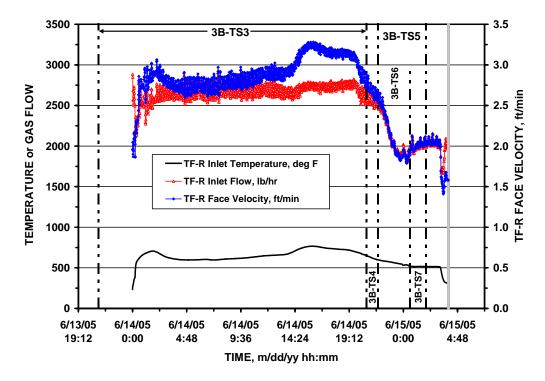


Figure 72 - Temperature, Syngas Flowrate, and Face Velocity at the Test Filter-Reactor Inlet during the NGC-OPT3B Campaign

#### 12.4.1.3 Syngas Mercury Measurements

The lignite-derived raw syngas was estimated to contain ~ 1.8 ppbv of elemental mercury (Hg0) or ~ 14.8  $\mu$ g/m3, if all the mercury in processed lignite (~ 0.06 ppmw) were released in the product syngas. Close to 200 gas, solid, and liquid samples were taken throughout the duration of the NGC-OPT3B campaign, as shown in Table 36 through Table 38. Solids included lignite feed (S-1), gasifier ash (S-5), secondary cyclone fines (S-6), and char samples from the first Sample Extraction and Conditioning (SE&C) Skid at the G-8 sampling location. Solid samples (Table 36 and Table 34) were analyzed mostly for ash to monitor gasifier performance, but some selected samples were also analyzed for mercury. Liquid (water) samples from the SE&C Skids at the G-8, G-13/G-14, and G-19 sampling locations were also analyzed for mercury to confirm viability of the sampling technique used (Table 38). Gas samples (Table 38) consisted mostly of dry samples (water removed by gas cooling to 40-50°F) taken at the various sampling points using gold traps. These traps were then analyzed for mercury off-line using a Nippon WA-4 Mercury Analyzer (~ 0.01  $\mu$ g/m3 detection limit). Other gas samples were taken via sampling cylinders at G-8 (to determine the overall composition and sulfur content of the raw syngas stream) and at G-14 (to confirm low-S content of the conditioned syngas and ensure that the SGB continued to perform well, Table 35).

Numerous Hg samples were taken during the third test segment (3B-TS3) when the gasifier was operating under steady state conditions and preferred operating conditions were being (or had been) established in the NGC Process section. As can be seen in Table 38, initial Hg analyses were very low, much lower than expected. At the G-8 location, it was suspected char on the Mott filter (maintained at 400°F) on the SE&C Skid #1 was interfering with the sampling. Water samples at G-8 contained very little Hg (about 1 ppb), ruling out the possibility that mercury was being lost to the water during gas cooling. At the G-13 location (CF-R outlet) where char is minimal or non-existent, it was suspected Hg could be removed by char within the CF-R vessel. Although the inlet and outlet CF-R temperatures were about 700°F, there were other locations within the vessel at much lower temperatures. Hg removal within the CF-R vessel would also explain the very low Hg analyses obtained at the G-14 location, where the conditioned syngas was essentially dust-free and desulfurized to very low levels.

Based on the results obtained a few options were determined to proceed. First, samples from the lignite feed were submitted for Hg analysis. The assays obtained, 0.061 and 0.090 ppmw (see 051263-070 and -089 in Table 38), confirmed the previous analysis that was performed in February 2005 on a composite sample. As discussed above, this Hg content in the feedstock could generate syngas mercury contents that would be at least 3 orders of magnitude higher than the detection limit of the Hg analysis method employed (~ 15 to 22  $\mu$ g/m<sup>3</sup>). [Note: additional post-test analyses on selected lignite feed samples indicated an average Hg content of ~ 0.084  $\mu$ g/g, as shown in Figure 73.] Therefore, it would not be helpful to switch to a different coal (such as washed Indian coal, which had a higher Hg content, about 0.14 ppmw). Second, an online analyzer (Tekran Mercury Vapour Analyzer 2537A) was quickly set up at the G-19 location that was much more sensitive than the off-line instrument. Simultaneously, we started sampling with gold traps at G-19, the TF-R outlet. Expecting to obtain a very low Hg content, we sampled for 90 minutes (see 051263-121 in Table 38). Immediately, the on-line analyzer indicated "high" Hg levels in the TF-R outlet syngas. Our Analytical Lab also informed us that the 90-min sample outranged their instrument, indicating Hg levels > 146 (Table 38). We were advised to reduce the sampling duration to 15 minutes or lower, and to discontinue using the on-line instrument for fear it would be "overwhelmed" and contaminated with mercury. Its use should be dedicated to sorbent injection periods, where the Hg level in the TF-R product syngas could be expected to be much lower.

It is possible that the gold traps at the G-8 and G-13 sampling locations were poisoned by sulfur in the syngas, preventing them from picking up any mercury. At these sampling locations,  $H_2S$  concentration was in the 1,000 to 1,200 ppmv range (Table 35). At G-14, where the syngas was desulfurized to a very high extent using the SGB, the gold traps could not have been poisoned. The fact

that no mercury could be measured at G-14 was probably caused by a malfunctioning temperature controller on the SE&C Skid #2. The Mott filter on this skid could not be maintained at the desired temperature of 400°F, and it was observed that this filter did not get heated beyond 277°F. Unfortunately, no residues could be collected from this sample train for Hg analysis to verify if any mercury removal took place at this location.

Five (5) gold trap samples were taken at the G-19 location prior to initiating sorbent injection. Two samples (#128 and #129 in Table 38) indicated 17.0 and 15.9  $\mu$ g/m<sup>3</sup>, which was well within the expected concentration range. One sample was not analyzed for fear its Hg content was too high because of much larger volume of gas. Unfortunately, the 2 samples that were taken immediately before sorbent injection measured lower Hg contents (5.9 and 4.9  $\mu$ g/m<sup>3</sup>). This discrepancy cannot be explained at this time.

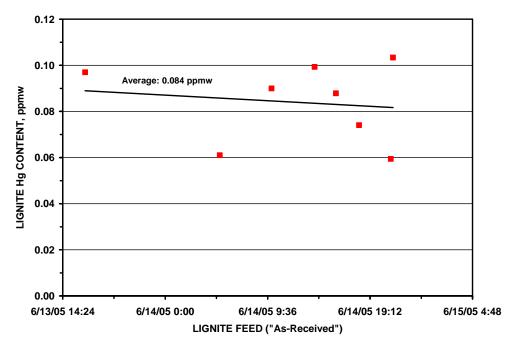


Figure 73 - Hg Content of Selected Samples from the Lignite Feed during the NGC-OPT3B Test Campaign

#### 12.4.1.4 Mercury Sorbent Performance Evaluation

As done in previous test campaigns, sorbent injection was performed in two phases, first at a higher sorbent injection rate to "pre-coat" the TF-R candles with sorbent before reducing the injection rate to the desired level and maintaining it for a given period of time. To evaluate mercury removal under TF-R optimum temperature for combined sulfur (H<sub>2</sub>S and COS) and halide (HCl) removal (i.e., 300°C or 572°F), the TF-R was operated under conditions that were nearly identical to the second test campaign. The TDA mercury sorbent, following activation and grinding at GTI as instructed by TDA, was selected as the "higher-temperature" sorbent. TDA sorbent was fed using the T-2107 Stage II Sulfur Sorbent Feeder. TDA sorbent injection was performed from ~ 21:45 on 06/14/05 to 02:00 on 06/15/05. To make a preliminary evaluation of the effect of the Stage II sulfur and halide sorbents on the performance of the Hg sorbent, an equal-weight mixture of the G-72E, G-92C, and nahcolite sorbents was fed (using the T-2108 Stage II Halide Sorbent Feeder) from ~ 00:35 to 02:00, concurrently with the TDA sorbent.

The Hg analyses at G-19 (TF-R outlet) during sorbent injection indicate some positive mercury removal results, particularly at such relatively high temperatures. During mercury sorbent injection, the Hg concentration in the TF-R product gas was measured at ~ 3.5 to 4  $\mu$ g/m<sup>3</sup>. Assuming a 16  $\mu$ g/m<sup>3</sup> mercury concentration in the TF-R inlet, then approximately 75% removal was achieved during injection of the mercury sorbent alone. When the sulfur and halide sorbents were injected concurrently with the mercury sorbent, mercury concentration in the TF-R outlet syngas slowly increased to ~ 8  $\mu$ g/m<sup>3</sup>, corresponding to about 50% removal. This was probably due to the fact that much less sulfur was available to interact with Hg. These data, summarized in Figure 74, can be used to develop appropriate projections of TDA's mercury removal sorbent performance at lower temperatures.

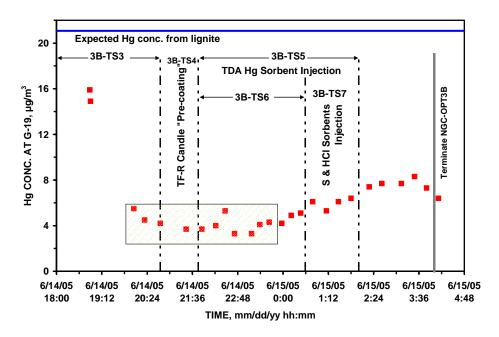


Figure 74 - Measured Hg Concentration at the TF-R Outlet during the Test Campaign #3 (NGC-OPT3)

As mentioned earlier, a plug developed in the spray quench tower, with the  $\Delta P$  gradually increasing to 10 psi or so through the course of the 6/14/05 evening and night. We started turning down the gasifier in the evening to reduce syngas flow and pressure drop across the plug, and that allowed enough time to proceed with the testing (determining the mercury level in the syngas and performing the "higher-temperature" sorbent injection tests). The NGC-OPT3B had to be terminated at ~ 04:00 on 6/15/05. The operating team brought the system down without any damage to the filter candles, and with a clean gasifier. Samples have been taken for post-test analyses to determine the nature of the plug so that it can be prevented in the future.

#### 12.4.1.5 Post-Test Analyses

In this test campaign we developed a clear approach to operating the facility and performing Hg sampling and analyses. However, additional testing should be performed to evaluate Hg removal at lower temperatures (350 to  $450^{\circ}$ F) using both the TDA and JM sorbents. Table 32 through Table 34 below provide additional analyses that were performed on solid samples collected post-tests. The Hg analyses for the coal feed, gasifier bottom ash, secondary cyclone fines, and CF-R fines confirm that ~ 98.4% of the Hg in the feed reported to the gas phase.

Proximate Analysis	(As received)	(As received)	(Dry basis)
I Tommute Timity 515	(115 10001/04)	w/SO <sub>3</sub> correction	w/SO <sub>3</sub> correction
Moisture, %	1.05	1.05	
Volatile Matter, %	6.20	6.20	6.27
Ash (750°C), %	74.57	69.05	69.78
Fixed Carbon, % (by difference)	18.18	23.70	23.95
Ultimate Analysis	(Dry basis)		
Ash (750°C), %	69.78	Mercury, µg/g	
		0.016	
Carbon, %	27.44		
Hydrogen, %	0.32		
Nitrogen, %	0.26		
Sulfur, %	2.35		
Oxygen, % (by difference)	B.D.L.		
Heating Value	(Dry basis)		
BTU/lb	4,040		
Screen Analysis			
	Retained on	Wt. %	
	6	0.0	
	12	0.0	
	20	0.0	
	40	0.0	
	60	0.2	
	80	0.2	
	100	0.4	
	140	1.9	
	200 230	5.1 3.4	
	230	6.3	
	325	6.3 5.5	
	PAN	77.0	
		//.0	
	Total	100.0	

Table 32 – NGC-OPT3 Equal Weight Composite Sample of Ten Secondary Cyclone Fines (Lab #'s 051263-085, -094, -098, -099, -105, -116, -119, -123, -126, & -130)

Proximate Analysis	(As received)	(As received)	(Dry basis)
		w/SO <sub>3</sub> correction	w/SO <sub>3</sub> correction
Moisture, %	0.51	0.51	
Volatile Matter, %	5.03	5.03	5.06
Ash (750°C), %	72.49	70.56	70.93
Fixed Carbon, % (by difference)	21.97	23.90	24.01
Ultimate Analysis	(Dry basis)		
Ash (750°C), %	70.93	Mercury, $\mu g/g < 0.010$	
Carbon, %	27.07		
Hydrogen, %	0.33		
Nitrogen, %	0.33		
Sulfur, %	0.80		
Oxygen, % (by difference)	0.54		
	0.0 .		
Heating Value	(Dry basis)		
BTU/lb	3,920		
Screen Analysis			
	Retained on	Wt. %	
	6	6.0	
	12	21.8	
	20	16.8	
	40	11.8	
	60	10.3	
	80	12.0	
	100	8.1	
	140	8.1	
	200	2.4	
	230	0.6	
	270	0.3	
	325	0.2	
	PAN	1.6	
	Total	100.0	

Table 33 - NGC-OPT3 Equal-Weight Composite Sample of Ten Gasifier Bottom Ash Fines (Lab #'s 051263-095, -100, -101, -106, -107, -117, -120, -124, -127, & -131)

Table 34 - NGC-OPT3 Equal-Weight Composite Sample of Four Conditioning Filter-Reactor Fines (T-2050 Drum 1 13:55 6/16/05, T-2050 Drum 3 6/16/05, T-2050 Drum 7 6/16/05, T-2153 Bottom 6/24/05)

Proximate Analysis	(As received)	(As received)	(Dry basis)
		w/SO <sub>3</sub> correction	w/SO <sub>3</sub> correction
Moisture, %	3.09	3.09	
Volatile Matter, %	11.10	11.10	11.45
Ash (750°C), %	60.20	56.81	58.62
Fixed Carbon, % (by difference)	25.61	29.00	29.93
Ultimate Analysis	(Dry basis)		I
Ash (750°C), %	58.62	Mercury, µg/g 0.028	
Carbon, %	35.50		
Hydrogen, %	0.42		
Nitrogen, %	0.34		
Sulfur, %	1.50		
Oxygen, % (by difference)	3.62		
Heating Value	(Dry basis)		
BTU/lb	5,050		
Screen Analysis			
	Retained on	Wt. %	
	6	0.6	
	12	0.4	
	20	0.4	
	40	1.0	
	60	1.0	
	80	1.0	
	100	0.6 2.3	
	140 200	2.3 2.7	
	230	1.7	
	230	2.3	
	325	2.3	
	PAN	83.1	
	Total	100.0	

Ref	Lab Sample ID	Sample Point	Date	Time	Test	H2	CO2	O2/Ar	N2	СО	CH4	Hexane	H2S	COS
	_	-			Segment							Plus	(ppmv)	(ppmv)
		<i></i>												
1	051263-073	G-8	6/14/2005	5:39	3B-TS3	13.2%	13.6%	0.70%	57.8%	12.29%	2.307%	0.017%	1040	83.6
2	051263-090	G-8	6/14/2005	9:30	3B-TS3	13.8%	13.6%	0.70%	56.6%	12.78%	2.385%	0.017%	1050	70.2
3	051263-091	G-8	6/14/2005	10:31	3B-TS3	13.8%	13.6%	0.69%	56.6%	12.75%	2.355%	0.017%	1090	73.5
4	051263-109	G-8	6/14/2005	11:30	3B-TS3	13.9%	13.5%	0.69%	56.4%	12.89%	2.377%	0.015%	1040	72.5
5	051263-110	G-8	6/14/2005	14:17	3B-TS3	14.1%	13.6%	0.69%	56.3%	12.79%	2.364%	0.016%	1080	74.6
6	051263-111	G-14	6/14/2005	14:20	3B-TS3	19.5%	19.4%	0.64%	52.9%	5.32%	2.204%	0.016%		0.11
7	051263-114	G-14	6/14/2005	16:30	3B-TS3	19.3%	19.1%	0.64%	52.9%	5.68%	2.298%	0.015%		0.47
8	051263-115	G-8	6/14/2005	16:35	3B-TS3	14.3%	13.7%	0.68%	55.9%	12.76%	2.441%	0.016%	1140	73.8
9	051263-180	G-8	6/14/2005	19:20	3B-TS3	14.1%	13.8%	0.69%	56.1%	12.70%	2.517%	0.016%	922	74.6
10	051263-149	G-14	6/14/2005	22:25	3B-TS5	14.4%	14.4%	0.66%	57.3%	10.73%	2.419%	0.014%		0.24
11	051263-152	G-8	6/14/2005	23:40	3B-TS5	13.9%	14.7%	0.66%	58.8%	9.52%	2.298%	0.014%	1200	65.4
12	051263-171	G-14	6/15/2005	2:35	3B-TS7	13.9%	15.2%	0.66%	59.1%	8.75%	2.353%	0.017%		0.14

 Table 35 - Summary of Batch Sample Gas Analyses during the NGC-OPT3B Test Campaign

					Test	As-received	Microwave	Mercury	
Ref	Lab Sample ID	Sample Point	Date	Time	Segment	Ash, wt%	Ash, wt%	micro-g/g	Comments
1	051263-004	S-6 (T-402)	6/9/2005	12:48		18.68	18.33	-	
2	051263-005	S-6 (T-402)	6/9/2005	13:45		42.51	40.87	-	
3	051263-007	S-6 (T-402)	6/9/2005	14:44		45.81	45.34	-	
4	051263-009	S-6 (T-402)	6/9/2005	15:41		50.02	49.82	-	
1	051263-014	S-6 (T-402)	6/13/2005	15:19	3B-TS1	31.48	31.19	-	
2	051263-016	S-6 (T-402)	6/13/2005	16:28	3B-TS2	34.62	34.24	-	
3	051263-017	S-6 (T-402)	6/13/2005	17:09	3B-TS2	34.95	34.66	-	
4	051263-020	S-6 (T-402)	6/13/2005	18:09	3B-TS2	36.54	36.87	-	
5	051263-022	S-4 (T-402)	6/13/2005	19:18	3B-TS2	43.24	42.29	-	
6	051263-024	S-4 (T-402)	6/13/2005	20:22	3B-TS2	51.70	51.50	-	
7	051263-028	S-4 (T-402)	6/13/2005	21:17	3B-TS2	53.82	52.82	-	
8	051263-031	S-4 (T-402)	6/13/2005	22:12	3B-TS3	56.24	55.46	-	
9	051263-035	S-4 (T-402)	6/13/2005	23:14	3B-TS3	60.44	59.02	-	
10	051263-040	S-4 (T-402)	6/14/2005	0:19	3B-TS3	61.52	61.71	-	
11	051263-044	S-4 (T-402)	6/14/2005	1:11	3B-TS3	62.22	60.31	-	
12	051263-048	S-4 (T-402)	6/14/2005	2:17	3B-TS3	55.74	55.77	-	
13	051263-052	S-4 (T-402)	6/14/2005	3:12	3B-TS3	63.71	63.05	-	
14	051263-056	S-4 (T-402)	6/14/2005	4:11	3B-TS3	65.84	65.19	-	
15	051263-066	S-4 (T-402)	6/14/2005	5:10	3B-TS3	66.48	66.02	-	
16	051263-074	S-4 (T-402)	6/14/2005	6:12	3B-TS3	66.66	65.70	-	
17	051263-078	S-6 (T-402)	6/14/2005	7:15	3B-TS3	-	67.22	-	
18	051263-079	S-6 (T-402)	6/14/2005	8:12	3B-TS3	-	68.79	-	
19	051263-084	S-6 (T-402)	6/14/2005	9:12	3B-TS3	-	70.50	-	
20	051263-085	S-6 (T-402)	6/14/2005	10:18	3B-TS3	-	71.50	-	
21	051263-094	S-6 (T-402)	6/14/2005	11:19	3B-TS3	-	71.94	0.015	
22	051263-098	S-6 (T-402)	6/14/2005	12:22	3B-TS3	-	73.40	-	
23	051263-099	S-6 (T-402)	6/14/2005	13:13	3B-TS3	-	75.16	-	
24	051263-105	S-6 (T-402)	6/14/2005	14:15	3B-TS3	-	74.67		
25	051263-116	S-6 (T-402)	6/14/2005	15:16	3B-TS3	-	74.63		
26	051263-119	S-6 (T-402)	6/14/2005	16:20	3B-TS3	-	73.77	-	
27	051263-123	S-6 (T-402)	6/14/2005	17:10	3B-TS3	-	73.92	-	
28	051263-126	S-6 (T-402)	6/14/2005	18:11	3B-TS3	-	73.48	-	
29	051263-130	S-6 (T-402)	6/14/2005	19:15	3B-TS3	-	73.34		
30	051263-136	S-6 (T-402)	6/14/2005	20:07	3B-TS3	-	71.57		
31	051263-141	S-6 (T-402)	6/14/2005	21:13	3B-TS4	-	69.44	-	
32	051263-144	S-6 (T-402)	6/14/2005	22:11	3B-TS5	-	69.90	-	
33	051263-150	S-4 (T-402)	6/14/2005	23:20	3B-TS5	-	68.33	-	
34	051263-160	S-4 (T-402)	6/15/2005	0:19	3B-TS5	-	67.56	-	
35	051263-162	S-4 (T-402)	6/15/2005	1:12	3B-TS5/3B-TS6	-	67.63	-	
36	051263-168	S-4 (T-402)	6/15/2005	2:16	3B-TS5/3B-TS6	-	65.83	-	
37	051263-174	S-4 (T-402)	6/15/2005	3:15	3B-TS7	-	64.92	-	

 Table 36 - Samples of Secondary Cyclone Fines during the NGC-OPT3 Test Campaign

					Test	As-received	Microwave	Mercury	
Ref	Lab Sample ID	Sample Point	Date	Time	Segment	Ash, wt%	Ash, wt%	micro-g/g	Comments
1	051263-001	S-5 (T-502)	6/9/2005	11:49		19.62	18.21	_	
2	051263-001	S-5 (T-502)	6/9/2005	12:32		15.62	15.03	-	
3	051263-003	S-5 (T-502)	6/9/2005	13:12		18.88	18.76	-	
4	051263-006	S-5 (T-502)	6/9/2005	14:20		30.57	30.09	-	
5	051263-008	S-5 (T-502)	6/9/2005	15:12		38.23	38.03	-	
1	051263-013	S-5 (T-502)	6/13/2005	14:40	3B-TS1	20.12	19.58	-	
2	051263-015	S-5 (T-502)	6/13/2005	15:39	3B-TS1	22.41	22.46	-	
3	051263-018	S-5 (T-502)	6/13/2005	16:44	3B-TS2	17.20	16.86	-	
4	051263-019	S-5 (T-502)	6/13/2005	17:38	3B-TS2	18.06	18.00	-	
5	051263-023	S-5 (T-502) S-5 (T-502)	6/13/2005	19:40	3B-TS2	22.22	22.05	-	
6 7	051263-025	S-5 (T-502) S-5 (T-502)	6/13/2005	20:38	3B-TS2	23.15	23.19	-	
8	051263-029	S-5 (T-502) S-5 (T-502)	6/13/2005	21:44	3B-TS3 3B-TS3	29.23	29.18	-	
8	051263-032 051263-036	S-5 (T-502) S-5 (T-502)	6/13/2005 6/13/2005	22:48 23:39	3B-183 3B-TS3	30.50 37.09	30.51 37.05	-	
9 10	051263-041	S-5 (T-502)	6/14/2005	0:50	3B-133 3B-TS3	38.51	38.47	-	
10	051263-041	S-5 (T-502)	6/14/2005	1:40	3B-133 3B-TS3	34.64	34.29	-	
11	051263-049	S-5 (T-502)	6/14/2005	2:41	3B-133 3B-TS3	37.56	37.50	-	
12	051263-053	S-5 (T-502)	6/14/2005	3:46	3B-133 3B-TS3	43.24	41.35		
13	051263-055	S-5 (T-502)	6/14/2005	4:44	3B-155 3B-TS3	51.08	50.88		
15	051263-067	S-5 (T-502)	6/14/2005	5:39	3B-155 3B-TS3	51.38	50.98		
16	051263-007	S-5 (T-502)	6/14/2005	6:42	3B-155 3B-TS3	54.10	53.31	-	
17	051263-086	S-5 (T-502)	6/14/2005	7:43	3B-TS3		44.66	-	
18	051263-087	S-5 (T-502)	6/14/2005	8:42	3B-TS3	-	50.84	-	
19	051263-088	S-5 (T-502)	6/14/2005	9:43	3B-TS3	-	60.20	-	
20	051263-095	S-5 (T-502)	6/14/2005	10:40	3B-TS3	-	65.32	0.005	
21	051263-100	S-5 (T-502)	6/14/2005	11:52	3B-TS3	-	69.64	-	
22	051263-101	S-5 (T-502)	6/14/2005	12:44	3B-TS3	-	71.21	-	
23	051263-106	S-5 (T-502)	6/14/2005	13:00	3B-TS3	-	76.75	-	
24	051263-107	S-5 (T-502)	6/14/2005	14:47	3B-TS3	-	74.93	-	
25	051263-117	S-5 (T-502)	6/14/2005	15:35	3B-TS3	-	80.43	-	
26	051263-120	S-5 (T-502)	6/14/2005	16:45	3B-TS3	-	75.73	-	
27	051263-124	S-5 (T-502)	6/14/2005	17:42	3B-TS3	-	73.90	-	
28	051263-127	S-5 (T-502)	6/14/2005	18:42	3B-TS3	-	73.27	-	
29	051263-131	S-5 (T-502)	6/14/2005	19:44	3B-TS3	-	61.20	-	
30	051263-137	S-5 (T-502)	6/14/2005	20:41	3B-TS3	-	71.31	-	
31	051263-142	S-5 (T-502)	6/14/2005	21:40	3B-TS4	-	76.18	-	
32	051263-145	S-5 (T-502)	6/14/2005	22:38	3B-TS5	-	69.24	-	
33	051263-151	S-5 (T-502)	6/14/2005	23:46	3B-TS5	-	69.81	-	
34	051263-161	S-5 (T-502)	6/15/2005	0:47	3B-TS5/3B-TS6	-	73.30	-	
35	051263-163	S-5 (T-502)	6/15/2005	1:54	3B-TS5/3B-TS6	-	76.99	-	
36	051263-169	S-5 (T-502)	6/15/2005	2:39	3B-TS7	-	79.51	-	
37	051263-175	S-5 (T-502)	6/15/2005	3:45	3B-TS7	-	73.48	-	

 Table 37 - Samples of Gasifier Ash during the NGC-OPT3 Test Campaign

1         00130400         CAUges         640206         1607-667         30	Ref	Lab Sample ID	Sample Point	Date	Time Period	Average Time	Test	Gas Hg Content	Solid/Liquid Hg Content
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$		-	-			(min)	Segment	(micro-grams/cu m)	(micro-grams/g)
$ \begin{array}{ c c c c c c } \hline 1 & 0.0126411 & C.4 (ga) & 0.0286 & 1718-17.38 & 20 &$									
•         •									
2         001204200         C4 (supp)         0412000         2012000         38.753         -0.2           3         001204301         C4 (supp)         0412000         22.44.352         38         38.753         -0.2           4         001204301         C4 (supp)         0412000         22.44.352         38         38.753         -0.2           6         00120430         C4 (supp)         0412000         22.44.352         38         38.753         -0.2           6         00120440         C4 (supp)         0412000         22.37.97         30         38.753         -0.2           9         00120440         C4 (supp)         0412000         002.24112         30         38.753         -0.2           10         00120440         C4 (supp)         0412000         00144224         39         38.753         -0.62           11         00120440         C4 (supp)         0412000         041400									
3         60120437         C412048         12632         126         12633         12633           4         60120441         C410grift         6112045         C410grift         641204         C410grift         641204           5         60120441         C410grift         6112045         2244.512         38         387.33         c482           7         66120441         C410grift         6112045         223.78607         30         387.33         c482           10         66120442         C410grift         6112045         223.78607         30         387.33         c482           11         6120440         C410grift         6112046         624.112         50         387.33         c482           11         6120446         C410grift         6122045         624.6224         53         387.33         c482           11         6120446         C410246         624.6224         53         387.33         c482           12         6120446         C410246         624.6244         64         387.33         c482           14         6120446         C410246         642.984         38         387.33         c482           14         6120446         <									
4         61204.30         LC (log at)         612085         16.29         38 - 783         -           5         601204.37         Lagis Ford         612085         22.34.232         38         38 - 783         -         -           6         61204.37         Lagis Ford         612085         22.37.4677         30         38 - 783         -         -           7         61204.30         GA (such)         6112085         22.37.4677         30         38 - 783         -         -           8         61204.30         GA (such)         6112085         02.27.4677         30         38 - 783         -         -         -           11         61204.46         GA (such)         6112085         02.34.344         36         38 - 783         -							3B-182 3B-T82	<0.02	<0.001
6         601246.40         Cold (valar)         611206.80         1244-25.22         38         38-733	4		LC (bag #1)				3B-TS3		0.097
7         00120430         C412008         123.00         30.733         -0.42           9         00120442         C412004         00120442         00         30.733         -0.42           10         00120442         C412005         00120442         30         30.733         -0.62           11         00120442         C412005         00120412         30         30.733         -0.62           12         00120440         C412005         00120412         30         30.733         -0.62           13         00120440         C412005         00124142         30         30.733         -0.62           14         00120440         C412005         00124434         56         30.733         -0.62           15         00120440         C412005         00124434         57         30.733         -0.62           16         00120440         C412005         00124443         64         30.733         -0.62           17         00120440         C412005         00124443         47         30.733         -0.62           18         00120447         C412005         00124445         47         30.733         -0.62           10         001210447	5	051263-033	G-8 (gas)	6/13/2005	22:44-23:22	38	3B-TS3	<0.02	0.004
8         61324.88         6.5 (ga)         613208         2.327.4607         9         33.733         -0.02           10         61324.84         6.4 (water)         614208         00.24112         9         33.733         -0.02           11         61324.84         6.4 (water)         614208         01.54.223         9         33.733         -0.02           12         61324.84         6.4 (water)         614208         01.54.223         9         33.733         -0.02           13         60324.84         6.4 (water)         614208         02.54.344         5         33.733         -0.02           14         61324.84         6.4 (water)         614208         03.84.84         38         33.733         -0.02           16         61324.84         6.4 (water)         614208         03.84.84         48         33.733         -0.02           10         61324.84         6.4 (water)         614208         03.84.84         47         33.733         -0.02           12         61324.84         6.4 (water)         614208         03.84.84         47         33.733         0.02           12         61324.84         6.4 (water)         614208         03.84.84         47 </td <td></td> <td>051263-034</td> <td></td> <td>6/13/2005</td> <td>23:30</td> <td>30</td> <td></td> <td></td> <td>0.004</td>		051263-034		6/13/2005	23:30	30			0.004
10         65126-842         67 (spa)         614/2005         00:224112         90         33:733         .0.02           11         65136-847         C4 (spa)         614/2005         01:24:123         39         33:733         .0.02           12         65136-847         C4 (spa)         614/2005         02:28:454         56         33:733         .0.02           15         65136-851         C4 (spa)         614/2005         02:28:454         56         33:733         .0.02           16         65136-851         C4 (spa)         614/2005         03:84:453         45         33:733         .0.02           17         65136-852         C4 (spa)         614/2005         03:84:453         45         33:733         .0.02           12         65136-862         C4 (spa)         614/2005         03:84:453         46         33:733         .0.02           13         65136-864         C4 (spa)         614/2005         04:84:46:1         77         33:733         .0.02           14         65136-846         C4 (spa)         614/2005         05:71         38:733         .0.02           12         65136-846         C4 (spa)         614/2005         57         38:733		051263-038	G-8 (gas)	6/13/2005	23:37-00:07		3B-TS3	<0.02	
11         65126343         65 (wher)         6142063         06124121         99         38733         cdd2           13         65126459         C4 (ga)         6142063         9234344         36         38733         cdd2           14         65126459         C4 (ga)         6142064         9234344         36         38733         cdd2           15         65126459         C4 (ga)         6142064         9234344         36         38733         cdd2           16         65126459         C4 (ga)         6142064         9354445         45         38733         cdd2           17         65126459         C4 (ga)         6142064         9354445         47         38733         cdd2           16         6126464         C4 (ga)         6142064         0438454         47         38733         cdd2           21         65126464         C4 (ga)         6442065         7         38733         cdd2           22         65126464         C4 (ga)         6442065         7         38733         cdd2           23         65126464         C4 (ga)         6442065         7         38733         cdd2           24         65126464         C41								-0.02	
		051263-042		6/14/2005	00:22-01:12	50	3B-153 3B-TS3	<0.02	
	12	051263-046	G-8 (gas)	6/14/2005	01:45-02:24	39	3B-TS3	<0.02	
								-0.02	
								<0.02	
	16	051263-054	G-8 (gas)	6/14/2005		38	3B-TS3	<0.02	
		051263-055			03:08-03:46	38	3B-TS3	-0.02	0.002
20         0612254060         C-31 gap)         0612005         0554041         47         38-TS3         0.02           21         061254061         C-31 water)         06142005         08540441         47         38-TS3         c.002           22         061254061         C-31 water)         06142005         0414005         47         38-TS3         0.008           23         061254065         C-31 water)         06142005         041446531         47         38-TS3         0.008           25         061254065         C-31 water)         06142005         38-TS3         0.01         0.01           27         061254061         Lippite Feed 4         06142005         38-TS3         0.01         0.01           31         061254076         C-31 gap)         06142005         06154770         45         38-TS3         0.01           32         061254076         C-31 gap)         06142005         075-6623         43         38-TS3         0.01           33         061254092         C-31 gap)         06142005         075-6623         43         38-TS3         0.01           34         061254092         C-31 gap)         0612005         55         38-TS3         0.01								<0.02	0.001
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$			G-13 (gas)			47		0.02	
1         0						47			
	22	051263-062 051263-063	G-8 (gas) G-8 (water)	6/14/2005 6/14/2005			3B-183 3B-T83	<0.02	0.001
	24	051263-064	G-13 (gas)	6/14/2005	04:44-05:31	47	3B-TS3	0.008	
			G-13 (water)		04:44-05:31	47			
	26	051263-068	Lignite Feed #3 Lignite Feed #4	6/14/2005			3B-TS3 3B-TS3		
	28		Lignite Feed #6		5:07				0.061
			G-13 (gas)					0.01	
$ \begin{array}{ c c c c c c } \hline 12 & 05124.6400 & C-13 (gas) & 06142005 & 0745-0623 & 43 & 331-TS3 & 0.01 & 014 & 015 & 014 & 015 & 014 & 015 & 014 & 015 & 014 & 015 & 014 & 015 & 014 & 015 & 014 & 015 & 014 & 015 & 015 & 014 & 015 $			G-13 (water)					0.01	
	32	051263-077	G-13 (water)	6/14/2005	06:15-07:42	87	3B-TS3	0.01	
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	33	051263-080	G-13 (gas)			43	3B-TS3	0.01	
	34						3B-TS3 3B-TS3	0.01	
			G-13 (water)					0.01	
	37	051263-089	Lignite Feed #7						0.090
	38	051263-092		6/14/2005		55	3B-TS3 3P TS3	0.01	0.036
	40	051263-096			10:40-11:25	45	3B-TS3	0.01	0.036
			G-13 (water)			45			0.018
	42					20		0.01	
	44	051263-103		6/14/2005	13:45-14:05	20	3B-TS3	0.01	0.002
	45				14:00				0.122 (dry basis) / 0.099
			G-14 (gas)					<0.01	0.009
						02			0.108 (dry basis) / 0.088
	49	051263-121	G-19 (gas)	6/14/2005	16:28-18:00		3B-TS3	> 146	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$						92			<0.001 0.091 (dry basis) / 0.074
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$						45		15.9	0.091 (dry basis) / 0.0/4
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	53	051263-129	G-19 (gas)	6/14/2005	18:46-19:02	16	3B-TS3	14.9	
	54		G-19 (gas)					not analyzed	
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	56	051263-135	G-19 (gas)					4.5	
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	57	051263-135	Lignite Feed #12	6/14/2005	21:09		3B-TS3		0.073 (dry basis) / 0.059
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	58	051263-138	G-19 (gas)			5	3B-TS4		
		051263-140	G-19 (gas)	6/14/2005	21:51		3B-TS4	3.7	
	61	051263-143	Lignite Feed #13	6/14/2005	21:21		3B-TS4		0.127 (dry basis) / 0.103
			G-19 (gas)						
	64		G-19 (gas)						
	65	051263-153	G-19 (gas)	6/14/2005	23:06-23:14	8	3B-TS5	3.3	
69         061263-157         G-19 (gas)         6152005         00:10-00:15         5         33-785         4.9           70         061263-158         G-19 (gas)         6152005         00:2-00:11         6         33-785         5.1           71         061263-159         G-19 (gas)         6152005         00:2-400:59         6         33-785         5.1           72         061263-159         G-19 (gas)         6152005         00:2-400:59         6         33-7858         5.1           73         061263-165         G-19 (gas)         6152005         00:4-40:59         6         33-7858-786         5.3           74         061263-165         G-19 (gas)         6152005         01:250-151         6         33-7858-758         5.3			G-19 (gas) G-19 (gas)						1
70         06123-138         G-19 (ggs)         6/152005         00:25-00:31         6         33-785         5.1           71         06123-159         G-19 (ggs)         6/152005         00:24-00:50         6         33-7858         6.1           72         06123-166         Lignite Feed #14         6/152005         08-14-00:50         38-7857.88-786         6.1           73         06123-165         G-19 (ggs)         6/152005         01:40-51:12         7         38-7857.88-786         5.3           74         06123-165         G-19 (ggs)         6/152005         01:26-01:12         6         38-7857.88-786         6.1	69	051263-157	G-19 (gas)	6/15/2005	00:10-00:15	5	3B-TS5	4.9	1
72         08128-164         Lignite Feed #14         6/152005         3B-TSS2B-TS6           73         05126-165         G-19 (gss)         6/152005         01:05-01:12         7         3B-TSS2B-TS6         5.3           74         05126-166         G-19 (gss)         6/152005         01:22-01:31         6         3B-TSS2B-TS6         5.3	70	051263-158	G-19 (gas)	6/15/2005	00:25-00:31	6	3B-TS5	5.1	1
73         051263-165         G-19 (gas)         6/15/2005         01:05-01:12         7         3B-TS5/3B-TS6         5.3           74         051263-166         G-19 (gas)         6/15/2005         01:25-01:31         6         3B-TS5/3B-TS6         6.1					00:44-00:50	6	3B-155/3B-TS6 3B-TS5/3B-TS6	6.1	1
74 051263-166 G-19 (gas) 6/15/2005 01:25-01:31 6 3B-TS5/3B-TS6 6.1	73	051263-165	G-19 (gas)	6/15/2005			3B-TS5/3B-TS6		
	74	051263-166	G-19 (gas)	6/15/2005	01:25-01:31		3B-TS5/3B-TS6		
75         0.612.63-167         G-1.9 (gas)         6/15/2005         01:45-01:51         6         38:TSS/38:TS6         6.4           76         0.612.63-110         Lignite Feed #15         6/15/2005         38:TSS/38:TS6         6.4					01:45-01:51	6		6.4	
77 051263-172 G-19 (gas) 6/15/2005 02:14-02:20 6 3B-TS7 7.4	77		G-19 (gas)			6			
78 051263-173 G-19 (gas) 6/15/2005 02:34-02:40 6 3B-TS7 7.7	78	051263-173	G-19 (gas)		02:34-02:40				
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$			G-19 (gas) G-19 (gas)	6/15/2005				7.7	1
81 051263-178 G-19 (gas) 6/15/2005 03:45-03:51 6 3B-TS7 7.3		051263-178	G-19 (gas)	6/15/2005	03:45-03:51		3B-TS7	7.3	1
82 051263-179 G-19 (gas) 6/15/2005 04:04-04:10 6 Terminate Test 6.4		051263-179	G-19 (gas)	6/15/2005	04:04-04:10	6	Terminate Test		
83 051263-184 Filter A (SE&C Skid #1) Post-test		051263-184	Filter A (SE&C Skid #1)						0.027
84 051263-185 Filter B (SE&C Skid #1) Post-test	84	051263-185	Filter B (SE&C Skid #1)	Post-test					0.005

 Table 38 - Summary of Mercury Analyses during the NGC-OPT3 Test Campaign

# **13. CONCLUSIONS AND RECOMMENDATIONS**

Three test campaigns were successfully completed as proof-of-principle demonstration of the NGC Process "filter-reactor" concept configuration in full integration with GTI's Flex-Fuel Test Facility. Extensive efforts in these tests were devoted to designing, installing, and validating state-of-the-art gas sampling equipment and instruments to meet the very stringent analytical needs of the program, involving measurements of concentrations of various compounds ranging from hundreds of parts-per-million (ppmv) to very low concentrations at the parts-per-billion (ppbv) level. The test program clearly demonstrated the flexibility of the Flex-Fuel Test Facility (gasifier/NGC Process section) to efficiently produce syngas from three different feedstocks, to condition the resulting raw syngas to meet the very stringent cleaning requirements of chemical synthesis applications (i.e., total S < 50 ppbv, total halides < 10 ppbv, particulate < 0.1 ppmw).

Consistent with the significance of the data developed in the PDU test program and the recommendations of conceptual process evaluations, GTI highly recommends the NGC Process development work enter into a process optimization phase. Specifically, four additional test campaigns are proposed prior to undertaking further scale-up work and ultimately commercial-scale demonstration. The objectives of these tests are to:

- Optimize key process parameters:
  - sorbent feed rates
  - Sorbent sizes and size distributions
  - process operating temperatures
  - inlet contaminant levels
- Explore the envelope of these parameters both separately and in an integrated configuration
  - Develop the necessary data to extract quantitative design parameters for scaling up the bulk HCl removal performance (in the Stage I barrier filter-reactor simultaneously with ash), and the combined removal of sulfur and halide species to ppbv levels in the Stage II barrier filterreactor.
  - Extract quantitative information (filter cake permeability, cake thickness, portion of ash reaching the filter elements, etc) from the pressure drop data (based on ash flow rate and size distribution to the CF-R and filter cake properties, such as density, re-entrainment rate, etc.)
- Conduct integrated testing to demonstrate NGC Process suitability for meeting "futuristic" IGCC fuel gas cleaning targets (sulfur, halides, mercury, and particulate). Futuristic is used to mean emissions from advanced natural gas-fired combined cycle systems. Ammonia will be monitored, but not controlled, in these test campaigns, which can be carried out in the existing NGC Process section in the Flex-Fuel Test Facility.
- Conduct integrated testing to demonstrate NGC Process suitability for meeting methanol synthesis cleaning targets (sulfur, halides, mercury, ammonia, and particulate). For these tests, a warm water scrubber column may be incorporated into the NGC Process to remove ammonia and control halides to less than 10 ppbv, eliminating the need for a Stage II halide sorbent. Commercial evaluations have shown this to be the best method for cleaning the methanol syngas stream in the co-production plant.

In addition to process optimization, the proposed campaigns will provide another opportunity to perform additional mercury removal testing (using both the lower temperature and higher temperature sorbents and potentially other promising Hg sorbents), further advancing the syngas mercury capture technology base. Other facets of the process will also be explored including bulk HCl removal at higher temperature, continuous removal of ash/spent halide sorbent fines from the CF-R, etc.

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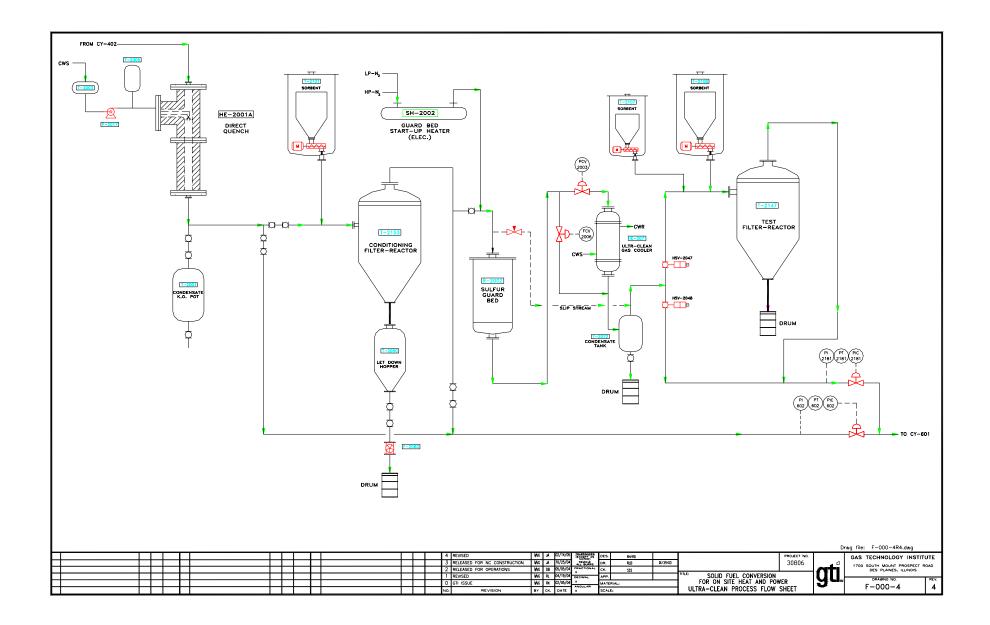
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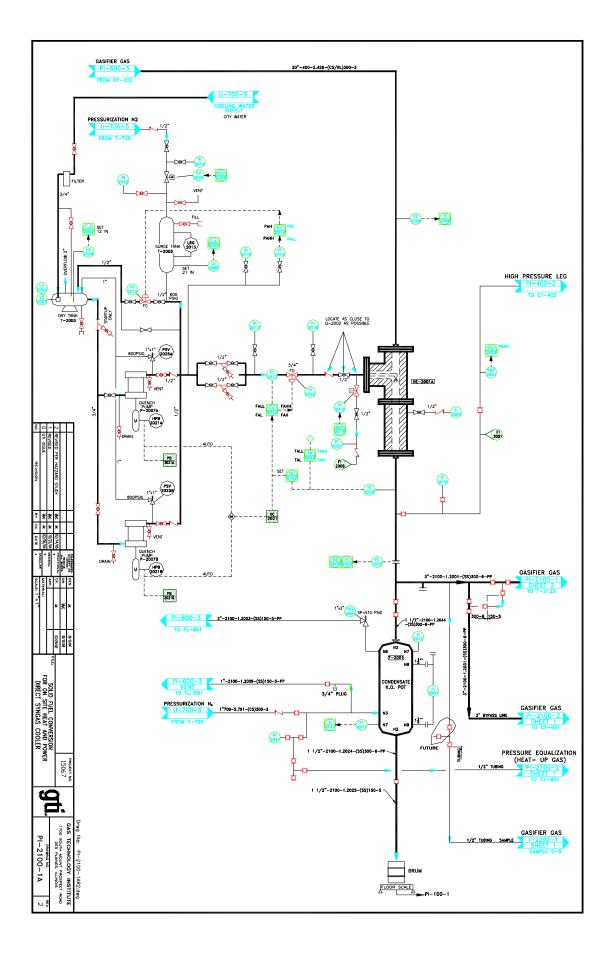
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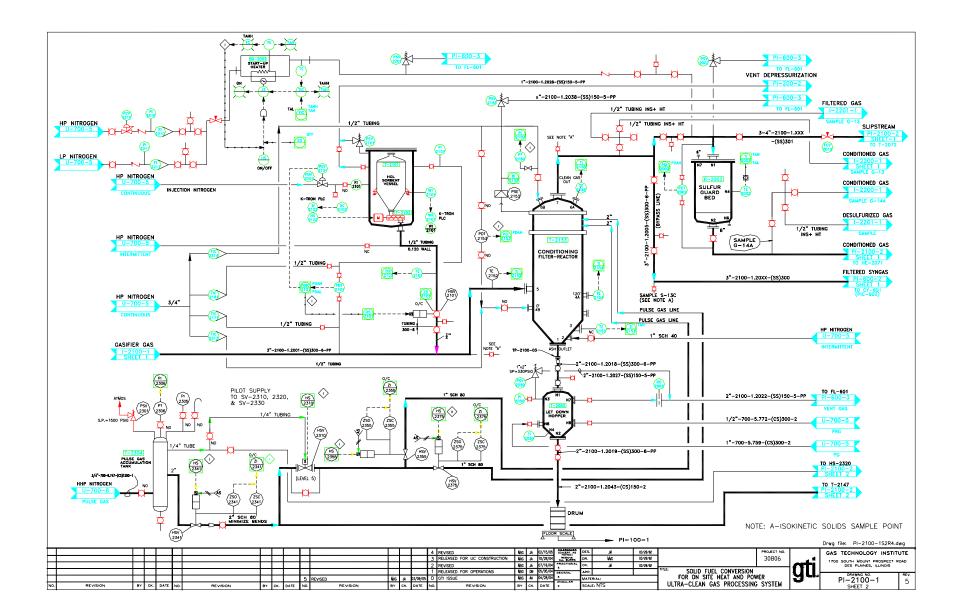
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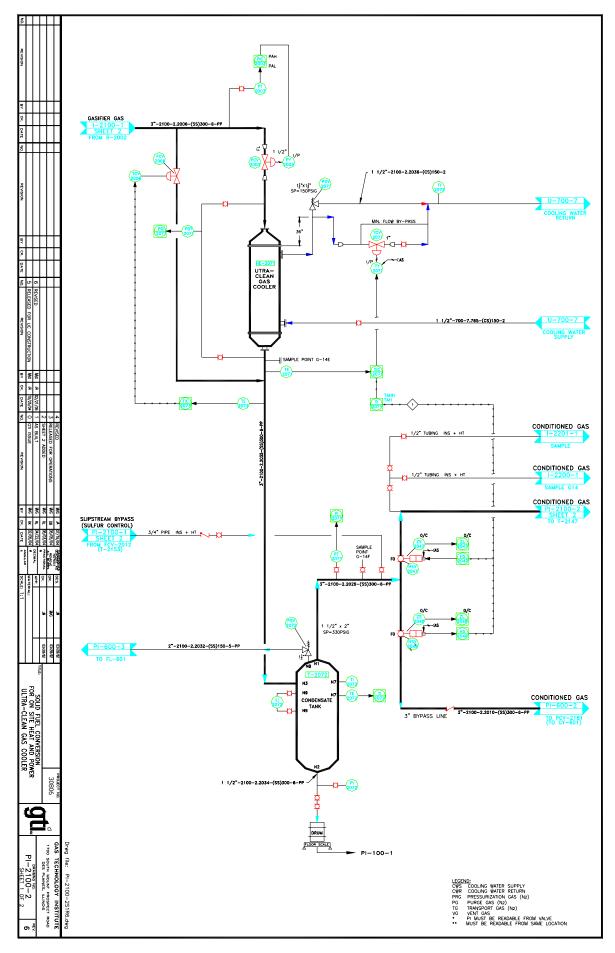
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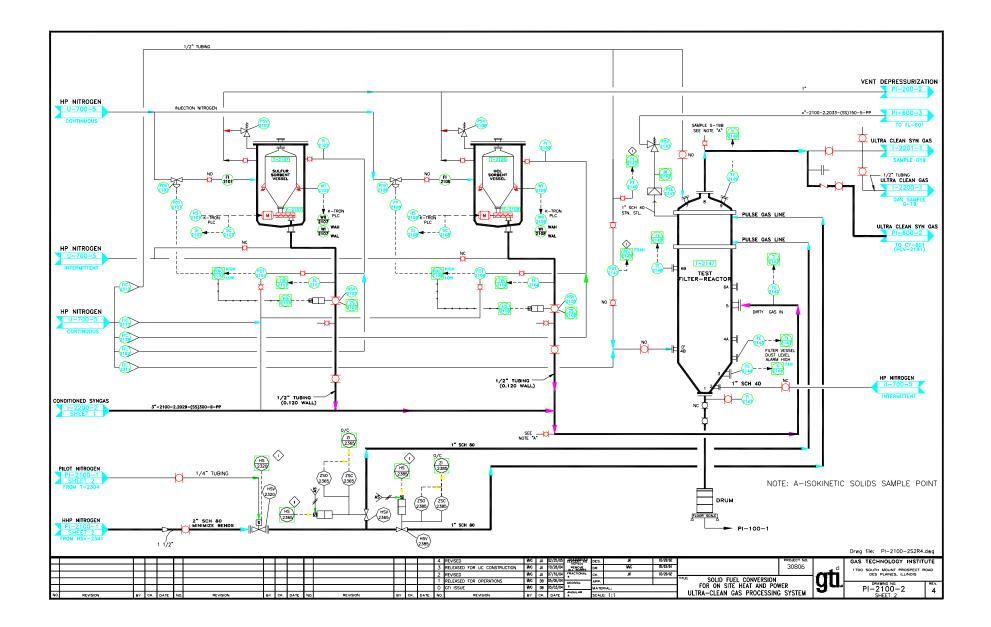
# 15. APPENDIX A: NGC PROCESS SECTION PIPING AND INSTRUMENTATION DIAGRAMS

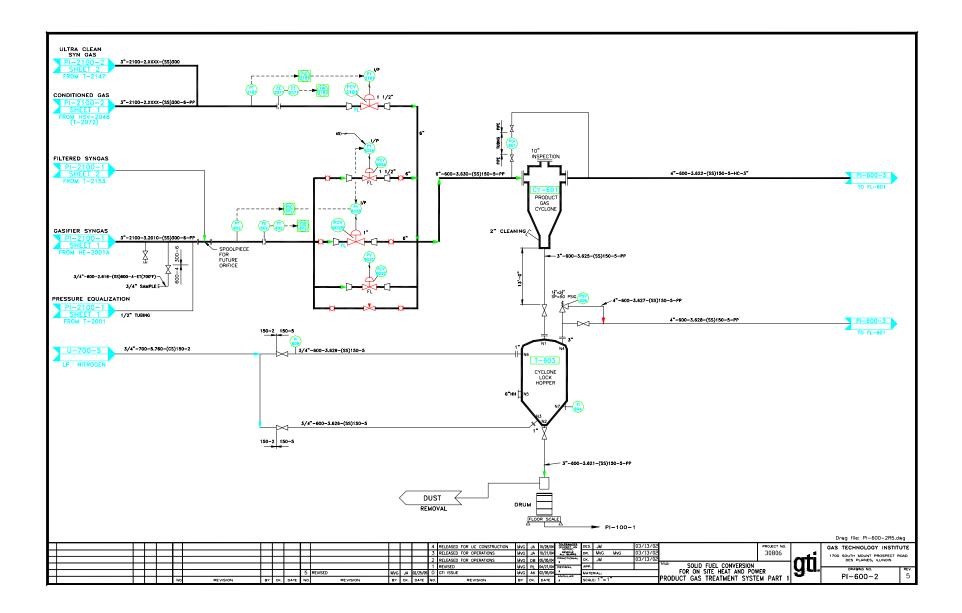


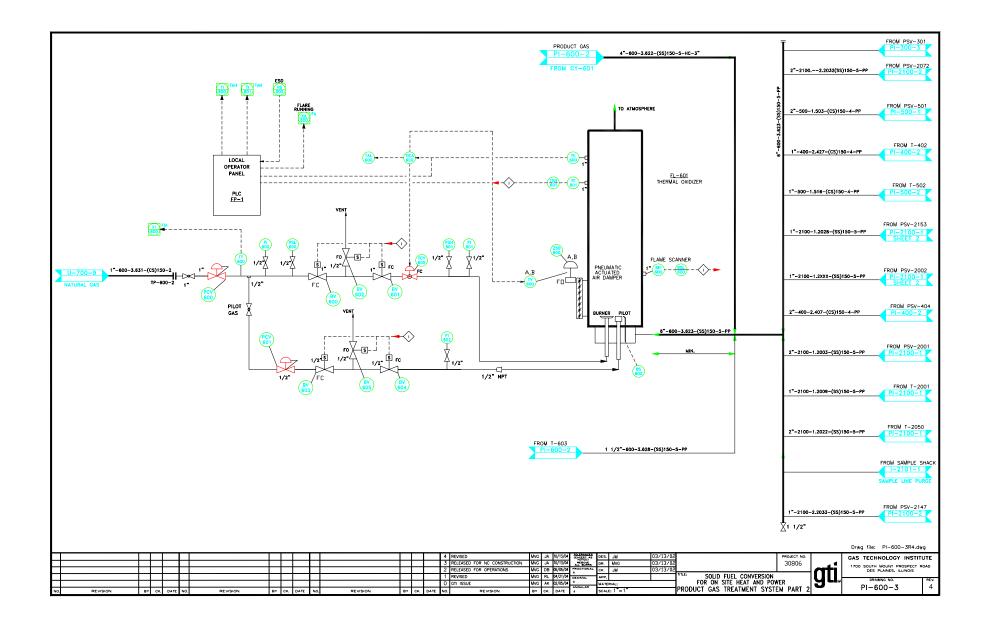


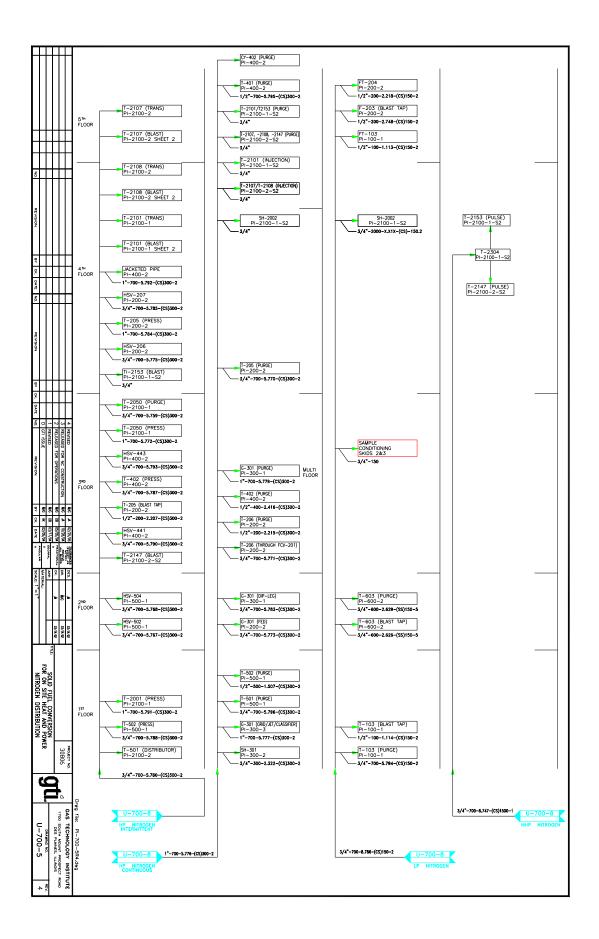




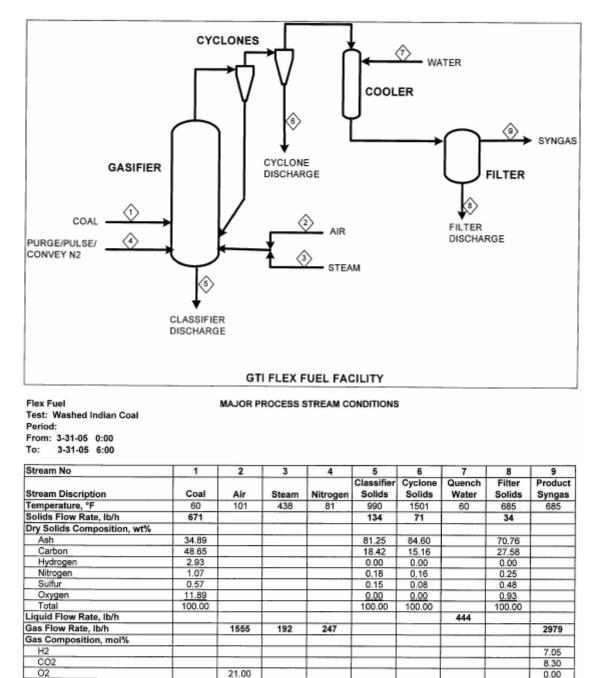








# **16. APPENDIX B: GASIFIER PERFORMANCE WITH WASHED INDIAN** COAL



100.00

100.00

100.00

0.00

100.00

79.00

0.00

100.00

N2

CO CH4 C2H6 C2H4

C6+

H2S

COS

H2O

Total

0.00

42.94

7.69 0.94 0.00 0.00

0.00

0.07

0.01

33.00

100.00

Flex Fuel Test Period	Washed	Indian Coal	OVERALL	PLANT MAS	S&HEAT BA	LANCE			
From	3/31/05	0:00							
То	3/31/05	6:00							
INPUT STREAMS		Carbon	Hydrogen	Oxygen	Nitrogen	Sulfur	Ash	Total	Btu/h
Coal Feed Air Feed		286.6	26.4	142.7	6.3	3.4	206.2	671.6	4.89E+06
Steam Feed			21.5	362.1 170.5	1192.7			1554.9	1.47E+04
N2 Feed			21.0	170.5	247.1			192.0 247.1	2.37E+05 1.19E+03
Water Feed			49.6	393.9	2.47.1			443.5	0.00E+00
Total Input		286.6	97.5	1069.2	1446.1	3.4	206.2	3109.1	5.15E+06
OUTPUT STREAM	IS								
Product Syngas		251.7	104.7	1132.8	1486.7	2.9		2978.8	4.09E+06
Classifier Discha		24.7	0.0	0.0	0.2	0.2	109.3	134.5	4.04E+05
2nd Cyclone Dis		10.8	0.0	0.0	0.1	0.1	60.6	71.6	1.94E+05
Filter Discharge Heat Loss		9.3	0.0	0.0	0.1	0.2	24.3	33.9	1.41E+05
Total Output		296.6	104.7	5422.0	4407.4				3.70E+05
rolai o'atpat		230.0	104.7	1132.8	1487.1	3.3	194.2	3218.8	5.20E+06
Out - In		10.0	7.1	63.6	41.0	-0.1	-12.0	109.7	4.96E+04
% Balance (Out/In)	)	103.5	107.3	105.9	102.8	96.9	94.2	103.5	101.0

(All units in lb/h unless otherwise noted)

# **GASIFIER OPERATING & PERFORMANCE PARAMETERS**

Gasifier Pressure, psig	256
Gasifier Bed Temperature, °F	1741
Coal Feed Rate, lb/h	671
Air Feed Rate, Ib/h	1555
Steam Feed Rate, lb/h	192
Steam/Carbon Ratio, Ib/Ib	0.67
Oxygen/Carbon Ratio, Ib/Ib	1.26
Gasifier Bed Density, lb/cu ft	32.1
Gasifier Bed Height, ft	9.5
Gasifier Superficial Velocity, ft/s	2.4
MAF Coal Gasification Intensity, lb/cu ft -hr	37.5
Dry & Purge N2 Free Syngas HHV, Btu/SCF	92.8
MAF Coal Conversion, %	88.2
Carbon Conversion, %	84.4

# Washed Indian Coal - Particle Size Distributions Test Period From 3/31/05 0:00 to 3/31/05 0:00

	COAL		FEED	CLASSIFIER SOLIDS		2° CYCLONE SOLIDS		FILTER SOLIDS	
		Differential	Cumulative	Differential	Cumulative	Differential	Cumulative	Differential	Cumulative
	Averaged Sieve	wť%,	wt%,	wt%,	wt%,	wt%,	wt%,	wt%,	wt%,
No	Opening, microns	Retained	Retained	Retained	Retained	Retained	Retained	Retained	Retained
6	4851	3.8	3.8	12.3	12.3	0.0	0.0	0.0	0.0
12	2516	10.6	14.4	27.3	39.6	0.0	0.0	0.0	0.0
20	1260	16.2	30.6	19.4	59.0	0.0	0.0	0.3	0.3
40	630	20.5	51.1	11.4	70.4	0.0	0.0	0.3	0.6
60	334	16.8	67.9	6.0	76.4	0.4	0.4	0.3	0.9
80	213	10.7	78.6	3.7	80.1	0.8	1.2	0.3	1.2
100	164	5.6	84.2	2.3	82.4	0.6	1.8	0.3	1.5
140	127	7.7	91.9	4.6	87.0	1.5	3.3	0.5	2.0
200	89	4.5	96.4	4.0	91.0	3.2	6.5	1.3	3.3
230	69	1.1	97.5	1.0	92.0	2.2	8.7	0.8	4.1
270	58	0.9	98.4	0.9	92.9	4.3	13.0	1.0	5.1
325	48	0.5	98.9	0.7	93.6	4.6	17.6	1.3	6.4
Pan	25	1.1	100	6.4	100.0	82.4	100.0	93.6	100.0
Total		100.0		100.0		100.0		100.0	
Avg Diame	ter*, microns	26	66	22	27	3	2	3	0

Note : Averaged particle diameter = Sauter mean Diameter =1/[summation (Wi/dpi)]

# **17. APPENDIX C: ANALYTICAL REPORT SAMPLES**

### **1. Gasifier Coal Feed**

Sample Login No: 051143-242

Air-Drv Moisture, %

Date: April 13, 2005

Sample Description: NGC-OPT2B composite of four "S1" feed coal bags #38, #39, #40, & #41

(Note: samples from bags 38 & 39 had visible condensation in the containers, samples 10.56 from bags 40 & 41 did not. This is the analysis of the entire composite.)

Proximate Analysis	(As received)	(As received) w/SO3 correction	(Dry basis) w/SO3 correction
Moisture, %	12.19	12.19	
Volatile Matter, %	25.34	25.34	28.86
Ash (750°C), %	30.73	30.63	34.89
Fixed Carbon, %	31.74	31.84	36.25

(by difference)

Ultimate Analysis	(Dry basis)
Ash (750°C), %	34.89
<b>C A A (</b>	10.15

Carbon, %	48.65
Hydrogen, %	2.93
Nitrogen, %	1.07
Sulfur, %	0.57
Oxygen, %	11.89
(by difference)	

Heating Value	<u>(Dry basis)</u>
BTU/lb.	8,300

### Analyst: NJP

# 2. Gasifier Bottom Ash

Sample Login No: 051143-243

Date: April 13, 2005

Sample Description: NGC-OPT2B composite of seven T-502's from 3/31/05 (Lab #'s 051143-205, 051143-207, 051143-210, 051143-212, 051143-216, 051143-218, & 051143-220)

Proximate Analysis	(As received)	(As received) w/SO3 correction	(Dry basis) w/SO3 correction
Moisture, %	0.27	0.27	
Volatile Matter, %	2.08	2.08	2.09
Ash (750°C), %	81.20	81.07	81.29
Fixed Carbon, %	16.45	16.58	16.62
(by difference)			
Ultimate Analysis	(Dry basis)		
Ash (750°C), %	81.29		
Carbon, %	18.43		
Hydrogen, %	< 0.01		
Nitrogen, %	0.18		
Sulfur, %	0.15		
Oxygen, %	B.D.L.		
(by difference)			
Heating Value	(Dry basis)		
BTU/lb.	2,570		

Analyst: NJP

# 3. Gasifier Secondary Cyclone Fines

### Sample Login No: 051143-244

Date: April 13, 2005

Sample Description: NGC-OPT2B composite of seven T-402's from 3/31/05 (Lab #'s 051143-206, 051143-208, 051143-211, 051143-213, 051143-217, 051143-219, & 051143-221)

Proximate Analysis	(As received)	(As received) w/SO3 correction	(Dry basis) <u>w/SO3 correction</u>
Moisture, %	0.39	0.39	
Volatile Matter, %	1.72	1.72	1.73
Ash (750°C), %	84.59	84.47	84.79
Fixed Carbon, %	13.30	13.42	13.48
(by difference)			
Ultimate Analysis	(Dry basis)		
Ash (750°C), %	84.79		
Carbon, %	15.19		
Hydrogen, %	< 0.01		
Nitrogen, %	0.16		
Sulfur, %	0.08		
Oxygen, %	B.D.L.		
(by difference)			
Heating Value	(Dry basis)		
BTU/lb.	2,130		

Analyst: NJP

# 4. Conditioning Filter-Reactor Fines

# Sample Login No: 051143-241 Sample Description: NGC-OPT2B T-2050 Dump # 14 11:33 4/01/05

Date: April 13, 2005

Proximate Analysis	(As received)	(As received) w/SO3 correction	(Dry basis) w/SO3 correction
Moisture, %	0.66	0.66	
Volatile Matter, %	4.22	4.22	4.25
Ash (750°C), %	71.64	70.30	70.76
Fixed Carbon, % (by difference)	23.48	24.82	24.99
Ultimate Analysis	(Dry basis)		
Ash (750°C), %	70.76		
Carbon, %	27.58		
Hydrogen, %	< 0.01		
Nitrogen, %	0.25		
Sulfur, %	0.48		
Oxygen, %	0.93		
(by difference)			
Heating Value	(Dry basis)		
BTU/lb.	4,030		

Analyst: NJP

5. Raw Syngas Bulk Composition and Sulfur Analyses (CF-R Inlet at G-8)

#### Major Component Gas Analysis By Gas Chromatography

Report Date: 8-Apr-05

Client Name: 15352.1.05

GTI Sample Number: 051143-204

Sample Description: NGC-OPT2 G8 Gas 3/31/2005 00:20

Date Analyzed: 7-Apr-05

Analyst: MAD

Component	Mol %	Det. Limit	Weight %
Helium		0.1%	
Hydrogen	10.5%	0.1%	0.78%
Carbon Dioxide	12.1%	0.03%	19.7%
Oxygen/Argon	0.73%	0.03%	0.88%
Nitrogen	63.9%	0.03%	66.0%
Carbon Monoxide	11.3%	0.03%	11.7%
Methane	1.41%	0.002%	0.836%
Ethane		0.002%	
Ethene		0.002%	
Ethyne		0.002%	
Propane		0.002%	
Propene		0.002%	
Propadiene		0.002%	
Propyne		0.002%	
i-Butane		0.002%	
n-Butane		0.002%	
1-Butene		0.002%	
i-Butene		0.002%	
trans-2-Butene		0.002%	
cis-2-Butene		0.002%	
1,3-Butadiene		0.002%	
i-Pentane		0.002%	
n-Pentane		0.002%	
neo-Pentane		0.002%	
1-Pentene		0.002%	
Hexane Plus	0.005%	0.002%	0.016%
Hydrogen Sulfide	0.090%	0.001%	0.114%
Carbonyl Sulfide	0.007%	0.001%	0.016%
Total	100.0%		100.0%
Calculated Real Gas I	Properties per ASI	TM D3588-98	
Temp. (°F) =	60.0	60.0	
Press. (psia) =	14.696	14.73	
Compressibility Factor [z] (Dry) =	0.99946	0.99946	
Compressibility Factor [z] (Sat.) =	0.99928	0.99927	
Relative Density (Dry) =	0.9361	0.9361	
Gross HV (Dry) (Btu/ft3) =	85.2	85.4	
Gross HV (Sat.) (Btu/ft3) =	83.8	84.0	
Wobbe Index =	88.1	88.3	

78.5 Net HV (Sat.) (Btu/ft3) = 77.1 77.3

Notes: All blank values are below detection limit N.A. - Not Analyzed

78.7

### 6. Syngas Bulk Composition and Sulfur Analyses (CF-R Outlet at G-13)

Net HV (Dry) (Btu/ft3) =

#### Major Component Gas Analysis By Gas Chromatography

Report Date: 24-Mar-05

Client Name: <u>15352.1.05</u>

GTI Sample Number: 051143-041

Sample Description: NGC-OPT2 G13 CFR Outlet 3/24/05 11:45

Date Analyzed: 24-Mar-05

Analyst: RJB

Component	Mol %	Det. Limit	Weight %
Helium		0.1%	
Hydrogen	10.0%	0.1%	0.74%
Carbon Dioxide	12.3%	0.03%	19.9%
Oxygen/Argon	0.70%	0.03%	0.83%
Nitrogen	65.6%	0.03%	67.5%
Carbon Monoxide	9.60%	0.03%	9.89%
Methane	1.76%	0.002%	1.04%
Ethane		0.002%	
Ethene		0.002%	
Ethyne		0.002%	
Propane		0.002%	
Propene		0.002%	
Propadiene		0.002%	
Propyne		0.002%	
i-Butane		0.002%	
n-Butane		0.002%	
1-Butene		0.002%	
i-Butene		0.002%	
trans-2-Butene		0.002%	
cis-2-Butene		0.002%	
1,3-Butadiene		0.002%	
i-Pentane		0.002%	
n-Pentane		0.002%	
neo-Pentane		0.002%	
1-Pentene		0.002%	
Hexane Plus	0.015%	0.002%	0.046%
Hydrogen Sulfide	0.0700%	0.001%	0.088%
Carbonyl Sulfide	0.0052%	0.001%	0.011%
Total	100.0%		100.0%

#### Calculated Real Gas Properties per ASTM D3588-98

Temp. (°F) =	60.0	60.0	
Press. (psia) =	14.696	14.73	
Compressibility Factor $[z]$ (Dry) =	0.99945	0.99945	
Compressibility Factor [z] (Sat.) =	0.99926	0.99926	
Relative Density (Dry) =	0.9396	0.9396	
Gross HV (Dry) (Btu/ft3) =	82.2	82.3	
Gross HV (Sat.) (Btu/ft3) =	80.7	80.9	
Wobbe Index =	84.8	85.0	
Net HV (Dry) $(Btu/ft3) =$	75.3	75.4	
Net HV (Sat.) (Btu/ft3) =	74.0	74.1	

Notes: All blank values are below detection limit N.A. - Not Analyzed

### 7. Conditioned Syngas Bulk Composition and Sulfur Analyses (TF-R Inlet at G-14)

#### Major Component Gas Analysis By Gas Chromatography

Report Date: 25-Mar-05

Client Name: <u>15352.1.05</u>

GTI Sample Number: 051143-060

Sample Description: NGC-OPT2 G14 TF-R Inlet 3/24/05 19:05

Date Analyzed: 24-Mar-05 Analyst: RJB

Component	Mol %	Det. Limit	Weight %
Helium		0.1%	
Hydrogen	12.6%	0.1%	0.94%
Carbon Dioxide	15.2%	0.03%	24.7%
Oxygen/Argon	0.68%	0.03%	0.81%
Nitrogen	63.6%	0.03%	66.0%
Carbon Monoxide	6.18%	0.03%	6.41%
Methane	1.78%	0.002%	1.06%
Ethane		0.002%	
Ethene		0.002%	
Ethyne		0.002%	
Propane		0.002%	
Propene		0.002%	
Propadiene		0.002%	
Propyne		0.002%	
i-Butane		0.002%	
n-Butane		0.002%	
1-Butene		0.002%	
i-Butene		0.002%	
trans-2-Butene		0.002%	
cis-2-Butene		0.002%	
1,3-Butadiene		0.002%	
i-Pentane		0.002%	
n-Pentane		0.002%	
neo-Pentane		0.002%	
1-Pentene		0.002%	
Hexane Plus	0.016%	0.002%	0.051%
Hydrogen Sulfide	0.000170%	0.000005%	0.00021%
Carbonyl Sulfide	0.000023%	0.000005%	0.00005%
Total	100.0%		100.0%

#### Calculated Real Gas Properties per ASTM D3588-98

Temp. (°F) =	60.0	60.0	
Press. (psia) =	14.696	14.73	
Compressibility Factor $[z]$ (Dry) =	0.99940	0.99940	
Compressibility Factor [z] (Sat.) =	0.99920	0.99920	
Relative Density (Dry) =	0.9324	0.9324	
Gross HV (Dry) (Btu/ft3) =	79.3	79.5	
Gross HV (Sat.) (Btu/ft3) =	77.9	78.1	
Wobbe Index =	82.1	82.3	
Net HV (Dry) (Btu/ft3) =	71.1	71.3	
Net HV (Sat.) (Btu/ft3) =	69.9	70.1	

Notes: All blank values are below detection limit N.A. - Not Analyzed

Thiophene detected at 0.68ppmv