

# **Wabash River Integrated Methanol and Power Production from Clean Coal Technologies (IMPPCCT)**

## **Quarterly Technical Progress Report No. 17**

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by

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## ABSTRACT

The Wabash River Integrated Methanol and Power Production from Clean Coal Technologies (IMPPCCT) project is evaluating integrated electrical power generation and methanol production through clean coal technologies. The project is under the leadership of ConocoPhillips Company (COP), after it acquired Gasification Engineering Corporation (GEC) and the E-Gas gasification technology from Global Energy in July 2003. The project has completed Phase I, and is currently in Phase II of development.

The two project phases include:

- I. Feasibility study and conceptual design for an integrated demonstration facility at Global Energy's existing Wabash River Energy Limited (WREL) plant in West Terre Haute, Indiana, and for a fence-line commercial embodiment plants (CEP) operated at Dow Chemical or Dow Corning chemical plant locations
- II. Research, development, and testing (RD&T) to define any technology gaps or critical design and integration issues.

The Phase I of this project was supported by a multi-industry team consisting of Air Products and Chemicals, Inc., Dow Chemical Company, Dow Corning Corporation, Methanex Corporation, and Siemens Westinghouse Power Corporation, while Phase II is supported by Gas Technology Institute, TDA Research Inc., and Nucon International, Inc.

The WREL integrated gasification combined cycle (IGCC) facility was designed, constructed, and operated under a project selected and co-funded under the Round IV of the United States Department of Energy's (DOE's) Clean Coal Technology Program. In this project, coal and/or other solid fuel feedstocks are gasified in an oxygen-blown, entrained-flow gasifier with continuous slag removal and a dry particulate removal system. The resulting product synthesis gas is used to fuel a combustion turbine generator whose exhaust is integrated with a heat recovery steam generator to drive a refurbished steam turbine generator. The gasifier uses technology initially developed by The Dow Chemical Company (the Destec Gasification Process), and now acquired and offered commercially by COP as the E-Gas technology.

In a joint effort with the DOE, a Cooperative Agreement was awarded under the Early Entrance Coproduction Plant (EECP) solicitation. GEC, and now COP and the industrial partners are investigating the use of synthesis gas produced by the E-Gas technology in a coproduction environment to enhance the efficiency and productivity of solid fuel gasification combined cycle power plants.

The objectives of this effort are to determine the feasibility of an EECP located at a specific site which produces some combination of electric power (or heat), fuels, and/or chemicals from synthesis gas derived from coal, or, coal in combination with some other carbonaceous feedstock. The project's intended result is to provide the necessary technical, economic, and environmental information that will be needed to move the EECP forward to detailed design, construction, and operation by industry.

The early entrance coproduction plant study conducted in Phase I of the IMPPCCT project confirmed that the concept for the integration of gasification-based (E-Gas) electricity generation from coal and/or petroleum coke and methanol production (Liquid Phase Methanol or LPMEOH™) processes was feasible for the coproduction of power and chemicals. The results indicated that while there are minimal integration issues that impact the deployment of an IMPPCCT CEP, the major concern was the removal of sulfur and other trace contaminants, which are known methanol catalyst poisons, from the synthesis gas (syngas). However, economic concerns in the domestic methanol market which is driven by periodic low natural gas prices and cheap offshore supplies limit the commercial viability of this more capital intensive concept.

The objective of Phase II is to conduct RD&T as outlined in the Phase I RD&T Plan to enhance the development and commercial acceptance of coproduction technology. Studies will address the technical concerns that will make the IMPPCCT concept competitive with natural gas-based systems in the commercial marketplace. Efforts in Phase II will investigate the cleanup of the syngas by removing contaminants, particularly sulfur species, to a level acceptable for the methanol synthesis catalyst, and reducing the cost of the current sulfur removal system such as via warm gas cleanup methods. Laboratory testing followed by on-site testing at WREL with bench-scale slipstream units will be conducted. Actual syngas produced by the facility will be evaluated at system pressure and temperature.

Two processes low in capital and operating costs will be investigated:

1. A regenerable activated carbon system developed by NUCON for removing sulfur species in the syngas to parts-per-billion (ppb) levels
2. A direct sulfur oxidation recovery process developed by TDA to remove sulfur species from sour syngas at warm gas temperatures

The two processes will also be evaluated in a hybrid configuration with the NUCON process downstream of the TDA process.

This report summarizes progress made during the period of October 1 – December 31, 2003.

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## ACRONYMS AND DEFINITIONS

<b>Acronym</b>	<b>Description</b>
ASU	Air Separation Unit
BFW	Boiler Feed-Water
CC	Combined Cycle (plant including only HRSG, CT & ST)
CEP	Commercial Embodiment Plant
CT	Combustion Turbine
CCT	Clean Coal Technologies
COP	ConocoPhillips Company
DOE	Department of Energy
EECP	Early Entrance Coproduction Plant
E-Gas	Title ConocoPhillips' gasification process
GEC	Gasification Engineering Corporation
HHV	Higher Heating Value in Btu/SCF
HRSG	Heat Recovery Steam Generator
HTHRU	High Temperature Heat Recovery Unit
IGCC	Integrated Gasification Combined Cycle
IMPPCCT Technologies	Integrated Methanol and Power Production from Clean Coal Technologies
LGTI	Louisiana Gasification Technology Incorporated
LOX	Liquid Oxygen
LPMEOH™	Liquid Phase Methanol (process)
MAC	Main Air Compressor
MDEA	Methyl-Di-Ethanol Amine (solvent)
MeOH	Methanol
MMBtu	Million British Thermal Units
Mt	Metric Ton
MTPD	Metric Ton Per Day
MW	Mega Watt
NETL	National Energy Technology Laboratory
NOx	Oxides of Nitrogen (symbol)
NPV	Net Present Value
O&M	Operating & Maintenance
PPMV	Parts Per Million Volume
PSE	Power Systems Engineering
RD&T	Research, Development & Test (plan)
SCF	Standard Cubic Foot
SFC	Synthetic Fuels Corporation
ST	Steam Turbine
Syngas	Synthesis Gas
TPD	Tons Per Day
WBS	Work Breakdown Structure (activities)
WREL	WABASH RIVER ENERGY LTD



## **1.0 BACKGROUND**

### **1.1 E-Gas Process Background**

The E-Gas gasification technology, recently acquired by ConocoPhillips Company (COP), is utilized at the Wabash River Energy Ltd., (WREL) facility located at Cinergy's Wabash River Generating Station in West Terre Haute, Indiana. WREL is a subsidiary of Global Energy, Inc., the previous owner of the E-Gas technology. COP, headquartered in Houston Texas, continues to develop and market the technology.

The E-Gas process features an oxygen-blown, continuous-slagging, two-stage, entrained-flow gasifier, which uses natural gas for start-up. Coal or petroleum coke is milled with water in a rod-mill to form slurry. The slurry is combined with oxygen in mixer nozzles and injected into the first stage of the gasifier, which operates at approximately 2600°F and 400 psi. A turnkey, Air Liquide, 2,060-ton/day low-pressure cryogenic distillation facility that WREL owns and operates, supplies oxygen of 95% purity.

In the first stage, slurry fuel undergoes a partial oxidation reaction at temperatures high enough to bring the coal's ash above its melting point. The fluid ash falls through a taphole at the bottom of the first stage into a water quench, forming an inert vitreous slag. The synthesis gas produced by this reaction then flows to the second stage, where additional coal slurry is injected. This coal is pyrolyzed in an endothermic reaction with the hot synthesis gas to enhance the heating value of the synthesis gas and to improve the overall efficiency of the process.

The synthesis gas then flows to the high-temperature heat-recovery unit (HTHRU), essentially a fire tube steam generator, to produce high-pressure saturated steam. After cooling in the HTHRU, particulates in the synthesis gas called char are removed in a hot/dry filter and recycled to the gasifier where the carbon content in the char is converted into synthesis gas. The synthesis gas is further cooled in a series of heat exchangers, is water scrubbed to remove the chloride, and is passed through a catalyst, which hydrolyzes carbonyl sulfide into hydrogen sulfide. Hydrogen sulfide is removed from the synthesis gas using a methyl-di-ethanol-based amine solvent in an absorber/stripper column process. The "sweet" synthesis gas is then moisturized, preheated, and piped over to the power block.

The key elements of the power block are the General Electric MS 7001 FA (GE 7 FA) high-temperature combustion turbine/generator, the heat recovery steam generator (HRSG), and the repowered steam turbine. The GE 7 FA is a dual-fuel turbine (synthesis gas for operations and No. 2 fuel oil for startup) that is capable of generating a nominal 192 MW when firing synthesis gas, about seven percent (7%) higher power production than the same turbine fired on natural gas. The enhanced power production is attributed to the increased mass flows associated with synthesis gas. Steam injection is used for control of nitrogen oxides called NO<sub>x</sub> within the combustion turbine. The required steam flow is minimal compared to that of conventional systems as the synthesis gas is moisturized at the gasification facility, by recovery of low-level heat in the process. The

water consumed in this process is continuously made up at the power block by water treatment systems, which clarify and further treat river water.

The HRSG for this project is a single-drum design capable of superheating 754,000 lb/hr of high-pressure steam at 1010°F, and 600,820 lb/hr of reheat steam at 1010°F when operating on design-basis synthesis gas. The HRSG configuration was specifically optimized to utilize both the gas-turbine exhaust energy and the heat energy made available in the gasification process. The nature of the gasification process in combination with the need for strict temperature and pressure control of the steam turbine led to a great deal of creative integration between the HRSG and the gasification facility. The repowered steam turbine produces 104 MW, which combines with the combustion turbine generator's 192 MW and the system's auxiliary load of approximately 34 MW to yield 262 MW (net) to the Cinergy grid.

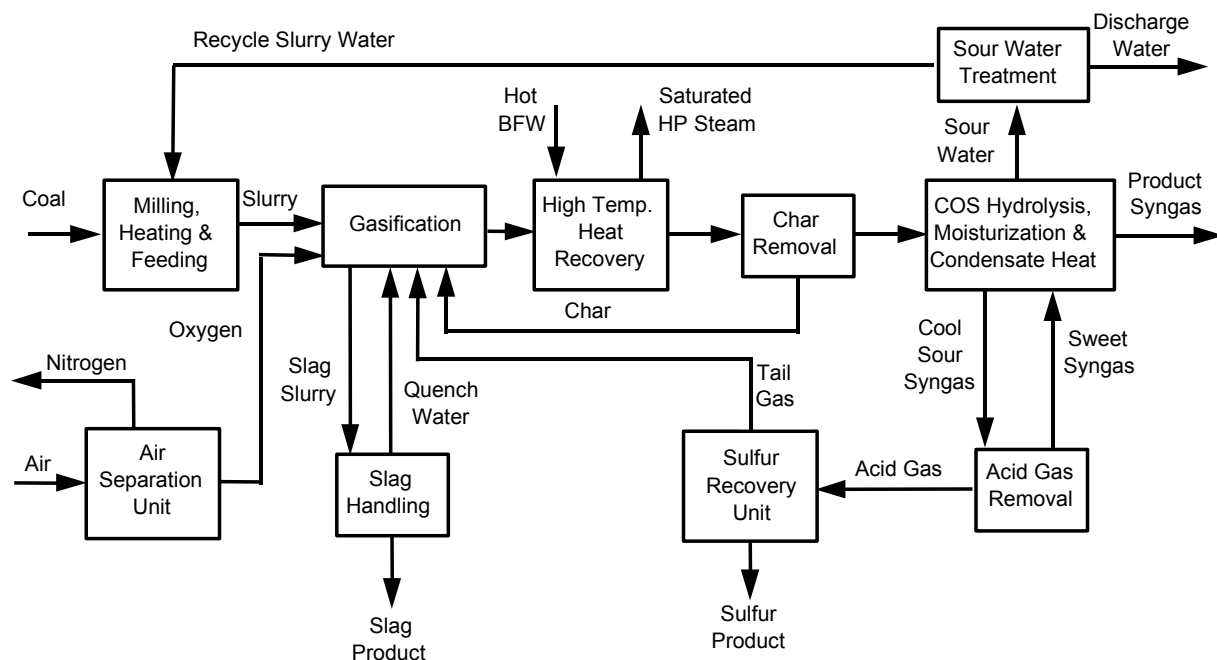
The Air Separation Unit (ASU) provides oxygen and nitrogen for use in the gasification process but is not an integral part of the plant thermal balance. The ASU uses services such as cooling water and steam from the gasification facilities and is operated from the gasification plant control room.

The gasification facility produces two commercial by-products during operation. Sulfur, which is ultimately removed as 99.99 percent pure elemental sulfur, is marketed to sulfur users. Slag is targeted as an aggregate in asphalt roads and as structural fill in various types of construction applications. In fact, the roads at the WREL facility have been top-coated with asphalt incorporating slag as the aggregate. Furthermore, at least two surrounding area sites have been audited, approved, and have used WREL-generated slag as structural fill under the Solid Waste Management Rules of Indiana. Another beneficial use of the slag by-product is as a fluxing agent during petroleum coke operation as this feed is typically deficient in mineral content required for proper slag fusion and flow. For this use, WREL has retained a reserve supply of slag generated from coal gasification.

The E-Gas process flow diagram presented in Figure 1.1.1 illustrates the features and components described in the above text. In Table 1.1.1, the WREL production statistics during the demonstration period of the Clean Coal Technology Program are presented in both English and Metric units. In Table 1.1.2, the WREL thermal performance variables are compared to the process design basis for both coal and petroleum coke feedstocks.

Please refer to the listing in Section 8.1 of this report for additional information on the Wabash River Coal Gasification Plant and the E-Gas technology.

**Figure 1.1.1: E-Gas Process Flow Diagram**



**Table 1.1.1 - WREL Gasification Production Statistics during the Demonstration Period of the Clean Coal Technology Program**

Production Variable	Production Year				
	1996	1997	1998	1999	2000
Gasifier Operation, Hrs	1,902	3,885	5,279	3,496*	3,406**
Dry Synthesis Gas Produced, GJ (MMBtu)	2,922,015 (2,769,683)	6,555,626 (6,213,864)	9,316,716 (8,831,011)	6,132,874 (5,813,151)	5,497,588 (5,210,984)
Coal Processed, Mt (Tons)	167,270 (184,381)	356,368 (392,822)	500,316 (551,495)	335,538 (369,862)	290,034 (319,703)

\* Three months of production were lost to the GE 7FA compressor failure & repair.

\*\* Three months of production were lost during commercial negotiations required when the WREL Facility transitioned to market-based operation.

**Table 1.1.2: Overall Thermal Performance of Gasification at WREL**

Performance Feature	Design	Actual Performance	
		Coal	Coke
NOMINAL THROUGHPUT, TPD	2550	2450	2000
Synthesis gas Capacity, MMBtu/hr	1780	1690 <sup>†</sup>	1690 <sup>†</sup>
Combustion Turbine, MW	192	192	192
Steam Turbine, MW	105	96	96
Aux. Power, MW	35	36	36
Net Generation, MW	262	261	261
Plant Efficiency, % (HHV)	37.8	39.7	40.2
Sulfur Removal Efficiency, %	>98	>99	>99

<sup>†</sup> Synthesis gas capacity referenced for coal and petroleum coke are the actual quantities fed to the combustion turbine when 192 MW (100%) of power generation occurs.

## 1.2 EECF Background Information

The request for Cooperative Agreement Proposals under the “Early Entrance Coproduction Plant (EECP),” Solicitation Number DE-SC26-99FT40040 was issued on February 17, 1999, by the United States Department of Energy.

The objective of this effort is to determine the feasibility of an EECF located at a specific site which produces some combination of electric power (or heat), fuels, and/or chemicals from synthesis gas derived from coal, or, coal in combination with some other carbonaceous feedstock. The scope of this effort includes:

1. Market analysis to define site-specific product requirements (i.e. products needed by market, market size, and price), process financials, feedstock availability, and feedstock cost;
2. System analysis to define feedstocks, feedstock preparation, conversion to synthesis gas, synthesis gas cleanup, and conversion of synthesis gas to market-identified products;
3. Preliminary engineering design of the EECF facility;
4. Preparation of a research, development, and testing (RD&T) Plan that addresses the technical uncertainties associated with eventual design, construction, and operation of the EECF;
5. Implementation of RD&T Plan;
6. Update of the preliminary engineering design; and

7. Update of the preliminary economic analysis.

Efforts under Solicitation No. DE-SC26-99FT40040 must support an EECP that at a minimum:

1. Is a single-train facility of sufficient size to permit scaling to commercial size with minimal technical risk;
2. Provides the capability of processing multiple feedstocks (must be capable of processing coal) and producing more than one product;
3. Is undertaken by an industrial consortium;
4. Reduces risk such that future coproduction plants may be deployed with no government assistance; and
5. Meets or exceeds environmental requirements and discusses the issue of carbon dioxide reduction by one or more routes, which include mitigation, utilization, and sequestration.

Using a focused RD&T Plan, the EECP Project will enhance the development and commercial acceptance of coproduction technology that produces high-value products, particularly those that are critical to our domestic chemical, fuel, and power requirements. The project will resolve critical knowledge and technology gaps on the integration of gasification and downstream processing to coproduce some combination of power, fuels and/or chemicals from coal or coal in combination with other carbonaceous feedstocks. The project's intended result is to provide the necessary technical, financial, and environmental information that will be needed to move the EECP forward to detailed design, construction, and operation by industry.

## 2.0 INTRODUCTION

The Wabash River Integrated Methanol and Power Production from Clean Coal Technologies (IMPPCCT) Project is a \$2,168,943 cooperative agreement awarded by the United States Department of Energy (DOE) to the former Gasification Engineering Corporation (GEC). The project is now under the leadership of ConocoPhillips Company (COP) after it acquired GEC and the E-Gas gasification technology from Global Energy in July 2003. The project evaluates the integration of gasification-based electrical generation and methanol production processes to determine the economic and technical feasibility of power and chemicals coproduction. A multi-industry team led by the previous GEC and consisting of Air Products and Chemicals, Inc., Dow Chemical Company, Dow Corning Corporation, Methanex Corporation, and Siemens Westinghouse Power Corporation performed and completed the Phase I IMPPCCT study. Phase II, which is currently in progress, is led by COP and is supported by Gas Technology Institute, TDA Research, Inc., and Nucon International, Inc.

The Wabash River IMPPCCT team is analyzing and developing a concept of methanol and power production based on the E-Gas gasification technology, now owned and licensed by COP, utilizing coal and other feedstocks. In the two-phase project, the team reviews and analyzes the domestic methanol market, examines the criteria needed and develop a financial model to study the economics of full-scale implementation of this gasification to power and methanol coproduction concept. Potential Dow Chemical and Dow Corning sites for the Commercial Embodiment Plant (CEP) were examined. Feasibility studies, testing and engineering, and economics of IMPPCCT based on addition of methanol production facilities at the Wabash River Energy Limited (WREL) Gasification Plant in West Terre Haute, Indiana are being developed to enable the commercialization of the gasification to power and methanol coproduction concept.

The vision of this project is to demonstrate the commercial viability of producing electric power, process energy (steam), and chemicals (methanol) from coal and other hydrocarbon feedstocks to satisfy the demands of at least two types and corresponding sizes of host chemical complexes. An efficient, low capital, integrated facility will convert the feedstock initially to synthesis gas and ultimately to electric power, process energy, and methanol with a series of reliable, commercially-proven, and environmentally-sound unit operations. The chemical products, required process energy, and at least a portion of the electric power will be delivered to the host chemical complex for further conversion to higher value products. Any products in excess of the requirements of the host chemical complex will be sold through readily accessible distribution networks. The CEP will be technically verified from the implementation of the Research, Development, and Testing (RD&T) Plan and commercially verified by an economic model.

### 3.0 EXECUTIVE SUMMARY

The Wabash River Integrated Methanol and Power Production from Clean Coal Technologies (IMPPCCT) project is evaluating integrated electrical power generation and methanol production through clean coal technologies. The project is conducted by a multi-industry team lead previously by Gasification Engineering Corporation (GEC). The project is now under the leadership of ConocoPhillips Company (COP) after it acquired GEC and the E-Gas gasification technology from Global Energy in July 2003. Phase I of this project was supported by Air Products and Chemicals, Inc., Dow Chemical Company, Dow Corning Corporation, Methanex Corporation, and Siemens Westinghouse Power Corporation, while the Phase II is supported by Gas Technology Institute (GTI), TDA Research Inc., and Nucon International, Inc. The project has completed Phase I, and is currently in Phase II of development. The two project phases include:

- I. Feasibility study and conceptual design for an integrated demonstration facility at Global Energy's existing Wabash River Energy Limited (WREL) plant in West Terre Haute, Indiana, and for a fence-line commercial embodiment plants (CEP) operated at Dow Chemical or Dow Corning chemical plant locations
- II. Research, development, and testing (RD&T) to define any technology gaps or critical design and integration issues.

The Wabash River Repowering Project, a joint effort between Wabash River Energy Limited (WREL) and Cinergy, was selected and co-funded under Round IV of the United States Department of Energy's (DOE's) Clean Coal Technology Program. In this project, coal and/or other solid fuel feedstocks are gasified in an oxygen-blown, entrained-flow gasifier with continuous slag removal and a dry particulate removal system. The resulting product synthesis gas is used to fuel a combustion turbine generator whose exhaust is integrated with a heat recovery steam generator to drive a refurbished steam turbine generator. The gasifier uses technology initially developed by The Dow Chemical Company (the Destec Gasification Process), and now offered commercially by COP as the E-Gas technology.

The demonstration project was completed in December 1999, having achieved all of its objectives. The facility built for this project is located at Cinergy Corporation's Wabash River Generating Station near West Terre Haute, Indiana.

The Wabash Repowering Project successfully demonstrated commercial application of the E-Gas coal gasification technology in conjunction with power generation. The combustion turbine generates 192 MW while the repowered steam turbine generates 104 MW. With the system's parasitic load of 34 MW, net power production is 262 MW, which meets the target goal. By the end of the demonstration period of the Clean Coal Technology Program, operating time had exceeded 18,000 hours, with over 5 million MW of power produced. The WREL Plant operates successfully on baseload dispatch in

the Cinergy power grid, and continues to operate as a privately owned facility after the demonstration period to supply synthesis gas to Cinergy.

Gasification is an environmentally superior means of utilizing domestic coal resources for power production. It also offers the opportunity to use lower quality, less expensive feedstocks such as petroleum coke. Petroleum coke operation was successfully tested at WREL as early as November 1997. Since August 2000, the facility has been operating on 100% petroleum coke feed. Over a million tons of fuel-grade petroleum coke has been processed, demonstrating the commercial viability of petroleum coke as the principle fuel for gasification.

Sulfur removal from the gasifier's solid feed is recovered and sold, as is the slag byproduct. Sulfur removal exceeds 97% resulting in sulfur oxides emissions of 0.1 lb/million Btu, which is far below regulatory requirements of 1.2 lb/million Btu. Particulate emissions are less than the detectible limit and nitrogen oxides emissions are 0.15 lb/million Btu, which meets the current target for coal-fired power generation plants. The WREL facility is the cleanest solid fuel-based power plants in the world.

In a joint effort with DOE, a Cooperative Agreement for IMPPCCT was awarded under the Early Entrance Coproduction Plant (EECP). GEC, and now COP, and the industrial partners are investigating the use of synthesis gas produced by the E-Gas technology in a coproduction environment to enhance the efficiency and productivity of solid fuel gasification combined cycle plants.

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The objective of Phase II is to conduct RD&T as outlined in the Phase I RD&T Plan to enhance the development and commercial acceptance of coproduction technology. Studies will address the technical concerns that will make the IMPPCCT concept competitive with natural gas-based systems in the commercial marketplace. Efforts in



Phase II will investigate the cleanup of the syngas by removing contaminants, particularly sulfur species, to a level acceptable for the methanol synthesis catalyst, and reducing the cost of the current sulfur removal system such as via warm gas cleanup methods. Laboratory testing followed by on-site testing at WREL with bench-scale slipstream units will be conducted. Actual syngas produced by the facility will be evaluated at system pressure and temperature.

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1. A regenerable activated carbon system developed by NUCON for removing sulfur species in the syngas to parts-per-billion (ppb) levels
2. A direct sulfur oxidation recovery process developed by TDA to remove sulfur species from sour syngas at warm gas temperatures

The two processes will also be evaluated in a hybrid configuration with the NUCON process downstream of the TDA process.

The Phase II testing projects were officially launched during the reporting period. GTI, who has been supporting TDA on developing their direct sulfur oxidation recovery process for natural gas application, agreed to provide operation personnel for the slipstream testing at WREL as well as to contribute funding to the project. GTI will become a subcontractor of COP, while TDA will be the subcontractor of GTI. Both Nucon and GTI, direct subcontractors of COP, were formally notified to start on their project under the IMPPCCT Phase II contract awarded COP by DOE. Subcontract documents between the parties are being drafted and reviewed. Project kickoff meetings were held at TDA and Nucon. Team members were apprised of the background and objectives of the IMPPCCT project, and were aligned on the objectives, schedule, milestones, and deliverables, as well as invoicing procedures, on the project. To ensure the RD&T is executed in an orderly and organized manner, COP prepared a Phase II RD&T Project Management Plan (PMP) with input from the subcontractors. The draft PMP was submitted to the DOE Project Manager for review at the end of the reporting period.

Both Nucon and TDA have successfully started on their projects. Nucon has begun on their laboratory testing. Initial experiments estimating the proper parameters of column diameter and length have been performed. Also a short series of experiments with impregnated activated carbon were conducted. Nucon has prepared a preliminary process design description and process and instrument diagram (P&ID). TDA has completed the design and procurement of most of the equipment necessary to modify their existing direct oxidation apparatus for the slipstream test at WREL and have begun construction of the unit. TDA is currently sizing some of the final components and completing the assembly of the apparatus.

The project successfully completed its first milestone, “Initiate construction of bench-scale, field-test units to remove sulfur and other trace contaminants present in synthesis gas”, during the reporting period.

## **4.0 ACTIVITIES**

The main activity during this reporting period was to get started on the testing projects identified for the Phase II investigation. Subcontractors were notified of the award. They in turn mobilized their work force, identified and assigned the proper personnel for their project, gathered their resources and equipment, and initiated the individual testing projects.

### **4.1 Project Management**

GTI, who has been supporting TDA on developing their direct sulfur oxidation recovery process for natural gas application, will provide operation personnel for the slipstream testing at WREL as well as contribute funding to the project. GTI will be a subcontractor of COP, while TDA will be a subcontractor of GTI.

Both Nucon and GTI, direct subcontractors of COP, were formally notified to start on their project under the IMPPCCT Phase II contract awarded COP by DOE, while a subcontract document with COP was being executed. GTI has in turn notified its subcontractor, TDA, of the award. Towards the end of the reporting period, draft subcontract agreements that include language on non-disclosure were sent to Nucon and GTI for review and comment.

A project kickoff meeting on TDA's slipstream testing project was held at TDA (Wheat Ridge, CO) on October 23, 2003 with a GTI representative participating. The project kickoff meeting with Nucon was postponed due to the unavailability of one of their key staff. The meeting was subsequently held at Nucon (Columbus, Ohio) on December 16, 2003.

To ensure the RD&T is executed in an orderly and organized manner, COP prepared a Phase II RD&T Project Management Plan (PMP). Nucon and TDA were asked to provide a detailed project schedule to be incorporated into the PMP.

### **4.2 Regenerable Carbon for Sulfur Polishing (Nucon)**

The objective of this study is to determine the feasibility and best means for the removal of sulfur-containing compounds from syngas using Nucon's impregnated regenerable activated carbon. The removal of the sulfur containing compounds - hydrogen sulfide ( $H_2S$ ), and carbonyl sulfide (COS) in this study - is critical in preventing the poisoning of the catalyst used to drive the chemical reaction to convert syngas to methanol. Laboratory tests will be conducted to determine the optimum sorbent and operating conditions. A slipstream unit will then be designed and constructed for on-site testing at WREL.

#### ***4.2.1 Conduct Laboratory Tests to Determine the Optimum Operating Condition for Slipstream Testing***

##### Analytical Methods

Analytical methods for H<sub>2</sub>S and COS in syngas mixture were developed by Nucon personnel prior to the start of the laboratory investigation.

##### Dynamic Adsorption Characteristics Determination

The adsorption characteristics of impregnated carbon are defined by the values of Mass Transfer Zone (MTZ) and Migration Rate (U). The mass transfer zone (MTZ) and migration rate (U) for both H<sub>2</sub>S and COS were determined as follows:

$$U = \frac{L}{t_{50}} \quad \text{and} \quad \text{MTZ} = U\Delta t$$

Where:

- U = rate of travel (in/min)
- L = bed depth (inches)
- t<sub>50</sub> = time to 50% breakthrough (min)
- MTZ = mass transfer zone (inches)
- Δt = time to saturation – time to breakthrough (min)

Initial evaluations of the metal oxide impregnated carbons were carried out in 2 in. long columns, having an internal diameter of 0.296 in. (see Table 4.2.1.1). The columns were stainless steel fitted with Swagelok fittings at both ends. The carbon was held in place using a fine wire screen at both ends of the carbon bed. The initial experiments were carried out using the 20 x 40 mesh granular carbon in order to maximize surface contact and to reduce consumption of the test gas. The test gas for these experiments was a mixture of 38.5% hydrogen (H<sub>2</sub>) and 68.5% carbon monoxide (CO), containing 25 parts per million (ppm) H<sub>2</sub>S, 25 ppm COS, and less than 1 ppm carbon dioxide (CO<sub>2</sub>). The low CO<sub>2</sub> concentration was used to minimize analytical interferences.

**Table 4.2.1.1:** Test Parameters for initial experiments evaluating metal oxide impregnated granular activated carbon (20 x 40 mesh) for the removal of hydrogen sulfide and carbonyl sulfide from a syngas mixture.

Bed Depth	2.0 inches (0.167 ft)
Internal Diameter	0.296 inches (*CA = 4.78 x 10 <sup>-4</sup> ft <sup>2</sup> )
Flow Rate	0.5 liter/min (0.0177 cu ft/min)
Velocity	45.09 ft/min
Residence Time	0.33 sec

\*CA, cross-sectional area

Four different impregnants in the activated carbon are under consideration at this time. These were selected based on previous work performed by Nucon, and by a literature search for other possible metal oxide impregnants with high efficiencies for the removal of sulfur compounds.

#### ***4.2.2 Design, Procure and Fabricate Slipstream Unit.***

The Nucon engineering department prepared a preliminary process description and Piping and Instrument Drawing (P&ID).

### **4.3 Direct Oxidation Sulfur Recovery (GTI/TDA)**

The overall objective of the testing project is to determine if TDA's direct oxidation sulfur recovery process can be used to desulfurize syngas produced by coal or petroleum coke gasification at the WREL plant. Preliminary data obtained in TDA's laboratory indicate that  $\text{H}_2\text{S}$  can be oxidized with high selectivity to elemental sulfur in the presence of large concentrations of  $\text{H}_2$  and  $\text{CO}$  with minimal (if any) oxidation of  $\text{H}_2$  or  $\text{CO}$ .

The preliminary tests were performed using a synthetic syngas made from laboratory gas cylinders. Thus, it is necessary to determine if there are any contaminants in actual produced syngas that might interfere with satisfactory performance of TDA's direct oxidation catalyst. This can only be determined by performing on-site tests using produced syngas as the feed.

#### ***4.3.1 Equipment Modification***

To determine if TDA's process is practicable for desulfurizing syngas from the WREL plant, a slipstream field test using TDA's automated catalyst test apparatus will be conducted. In order to do this however, TDA must: 1) modify their existing bench-scale direct oxidation unit for high pressure and high flowrate operation, 2) perform shakedown bench-scale tests of the apparatus at TDA with simulated syngas that has a composition that closely matches that of the WREL plant, and 3) transport the bench-scale unit to the WREL plant and perform desulfurization tests with a slipstream of syngas from the gasifier. Activities conducted in the reporting quarter were mainly focused on modifying the bench-scale unit.

## **5.0 RESULTS AND DISCUSSION**

Notice for approval of the Continuation Application of the project into Phase II to conduct RD&T was received from DOE in September 2003. The main area being pursued in RD&T is in synthesis gas contaminant removal. Potential technologies to be evaluated include Nucon's regenerable activated carbon and TDA's direct sulfur oxidation process to remove hydrogen sulfide and other sulfur species. Laboratory and slipstream testing at WREL are planned in the evaluation of both technologies. Small-scale slipstream testing at WREL, using actual synthesis gas being produced, is the preferred mode of testing. Laboratory testing will be conducted prior to the on-site slipstream testing to determine the optimum operating condition for the slipstream unit.

### **5.1 Project Management**

Both Nucon and GTI, subcontractors of COP, were formally notified to start on their project under Phase II of the IMPPCCT Project, while subcontract documents with COP were being executed. GTI, in turn, notified its subcontractor, TDA, to initiate work. Near the end of the reporting period, draft subcontract agreements that included specific non-disclosure agreement language were sent to Nucon and GTI for review and comment.

A project kickoff meeting on TDA's slipstream testing project was held at TDA (Wheat Ridge, CO) on October 23, 2003 with a GTI representative participating. The project kickoff meeting with Nucon was postponed due to the unavailability of one of their key staff members. The meeting was subsequently held at Nucon (Columbus, Ohio) on December 16, 2003. In both kickoff meetings, the COP Project Director reviewed with team members the background and objectives of the IMPPCCT Project. A Power Point presentation from the meetings is included in the Appendix. Team members were aligned on the objectives, schedule, milestones, and deliverables, as well as invoicing procedures, for the project.

To ensure that RD&T is executed in an orderly and organized manner, COP prepared a Phase II RD&T Project Management Plan (PMP). Nucon and TDA provided revised project schedules that were incorporated into the PMP. The draft PMP was submitted to the DOE Project Manager for review at the end of the reporting period.

### **5.2 Regenerable Carbon for Sulfur Polishing (Nucon)**

The objective of this study is to determine the feasibility and best means for the removal of sulfur-containing compounds from syngas using Nucon's regenerable impregnated activated carbon. The removal of the sulfur containing compounds - hydrogen sulfide ( $\text{H}_2\text{S}$ ), and carbonyl sulfide ( $\text{COS}$ ) in this study - is critical in preventing the poisoning of the catalyst used to drive the chemical reaction to convert syngas to methanol.

Laboratory tests will be conducted to determine the optimum sorbent and operating conditions. A slipstream unit will then be designed and constructed for on-site testing at WREL.

### ***5.2.1 Conduct lab tests to determine optimum operating condition for slipstream unit.***

#### Analytical Methods

Hydrogen sulfide and carbonyl sulfide in syngas are detected using a Matheson-Kitagawa precision gas detector pump (Model No. 8014-400A) with the appropriate detector tubes. Initial breakthrough of the sulfur gases is monitored by passing the gas stream exiting the carbon column over moist filter paper impregnated with lead acetate. At the first sign of discoloration, samples are drawn to determine H<sub>2</sub>S and COS concentrations at initial breakthrough and followed until the carbon bed is saturated as demonstrated by outlet concentrations of 25 ppm for H<sub>2</sub>S and COS. Hydrogen sulfide is detected using Matheson-Kitagawa detector tube no.s 120U (0.2 to 6 ppm H<sub>2</sub>S) and 120SD (1-60 ppm H<sub>2</sub>S). Carbonyl sulfide is detected with the Matheson-Kitagawa tube no. 239S (5-60 ppm COS). Previous experiments indicated that CO<sub>2</sub> could interfere with the measurement of COS. Therefore, a syngas mixture with less than 1 ppm CO<sub>2</sub> was used for the preliminary lab tests. Actual COS concentrations are determined by subtracting the COS tube response at the beginning of each column run from the values observed after H<sub>2</sub>S breakthrough. Additionally, Gastec COS detector tubes are used to watch for COS saturation, as they use a different reaction that is not subject to interference by CO<sub>2</sub>.

#### Dynamic Adsorption Characteristics

Initial evaluations of the metal oxide impregnated carbons were carried out in 2 in. long columns having an internal diameter of 0.296 in. (see Table 4.2.1.1). The columns were stainless steel fitted with Swagelok fittings at both ends. The carbon was held in place using a fine wire screen at both ends of the carbon bed. The initial experiments were carried out using the 20 x 40 mesh granular carbon in order to maximize surface contact and to reduce consumption of the test gas.

Four different impregnants in activated carbon are under consideration at this time. These were selected based on previous work performed by Nucon, and by a literature search for other possible metal oxide impregnants with high efficiencies for the removal of sulfur compounds. While all of the carbons exhibited a similar behavior and efficiency (8 to 11%) in initial tests using hydrogen sulfide in nitrogen, tests using the syngas mixture will be the standard for all recommendations.

To date, all studies have been performed using 20 x 40 mesh carbon to enable experiments to be conducted in a timely fashion. Additional impregnations of 1.5 mm and 3.0 mm pelletized carbon have been completed and small test columns assembled to determine the effect of particle size on over-all efficiency and aid in selecting the best carbon size for the application. Total capacity, mass transfer zone, migration rates were determined for each of the metal oxide impregnants, as well as breakthrough times and

time to saturation. The test gas for these experiments was a mixture of 38.5% hydrogen (H<sub>2</sub>) and 68.5% carbon monoxide (CO), containing 25 ppm H<sub>2</sub>S, 25 ppm COS, and less than 1 ppm CO<sub>2</sub>. Test results from carbon with the four impregnants using syngas is shown in Tables 5.2.1.1 and 5.2.1.2:

**Table 5.2.1.1 - H<sub>2</sub>S Data for Metal Oxide Impregnated Carbons (20 x 40 mesh)**

Impregnant	MO1	MO2	MO3	MO4
L (in)	2.0	2.0	2.0	2.0
t <sub>b</sub> (min)	135	192	102	221
t <sub>s</sub> (min)	210	267	315	296
t <sub>50</sub> (min)	170.8	231.7	200.3	253.0
Δt (min)	75	75	213	75
U (in/min)	0.012	0.009	0.01	0.008
MTZ (in)	0.9	0.68	2.13	0.6
Capacity (mg H <sub>2</sub> S/g)	23	33	18	38

**Table 5.2.12 – COS Data for Metal Oxide Impregnated Carbons (20 x 40 mesh)**

(Note that these are “apparent” values, as the conversion of COS to H<sub>2</sub>S will occur as long as reactive metal ions are present.)

Impregnant	MO1	MO2	MO3	MO4
L (in)	2.0	2.0	2.0	2.0
t <sub>b</sub> (min)	135	192	165	221
t <sub>s</sub> (min)	210	252	255	281
t <sub>50</sub> (min)	150.8	209.9	181.9	208.4
Δt (min)	75	60	90	60
U (in/min)	0.015	0.01	0.011	0.01
MTZ (in)	0.3	0.6	0.99	0.6
Capacity (mg COS/g)	41	59	50.6	58

L = bed depth

t<sub>b</sub> = time to breakthrough

t<sub>s</sub> = time to saturation

t<sub>50</sub> = time to 50% breakthrough

Δt = time to saturation – time to breakthrough

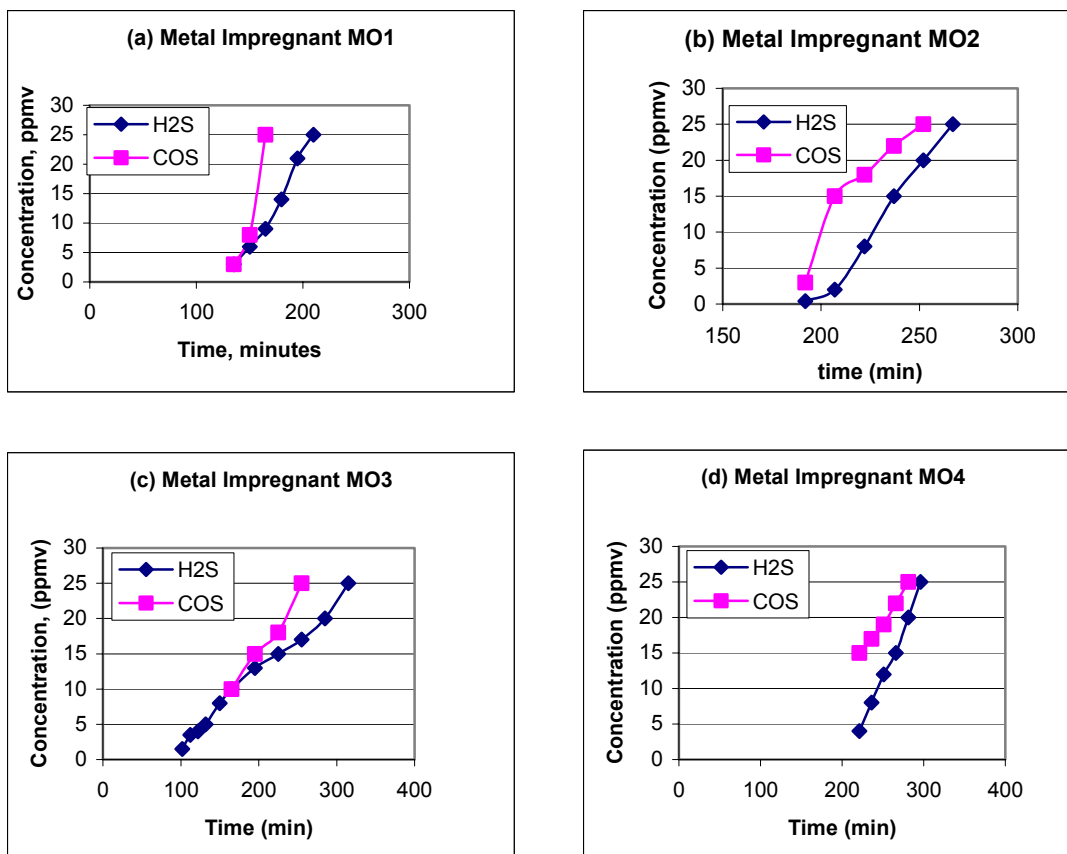
U = rate of travel

MTZ = mass transfer zone

Results are shown graphically as in Figures 5.2.1.1(a-d) :

**Figure 5.2.1.1(a-d)**

**Absorption Characteristics of Metal Oxide Impregnated Carbons**



**5.2.2 Design, Procure and Fabricate Slipstream Unit.**

The Nucon engineering department is making a drawing of the preliminary process design for the slipstream unit. The reactor column for the slipstream tests is tentatively sized to be 4" diameter. The length of the column will be determined by the lab test results. The remainder of the system will be constructed with 3/8" tubing. Stainless steel will be used. The appropriate flow, temperature and pressure instruments will be supplied. The preliminary P&ID is shown in Figure 5.2.2.1.



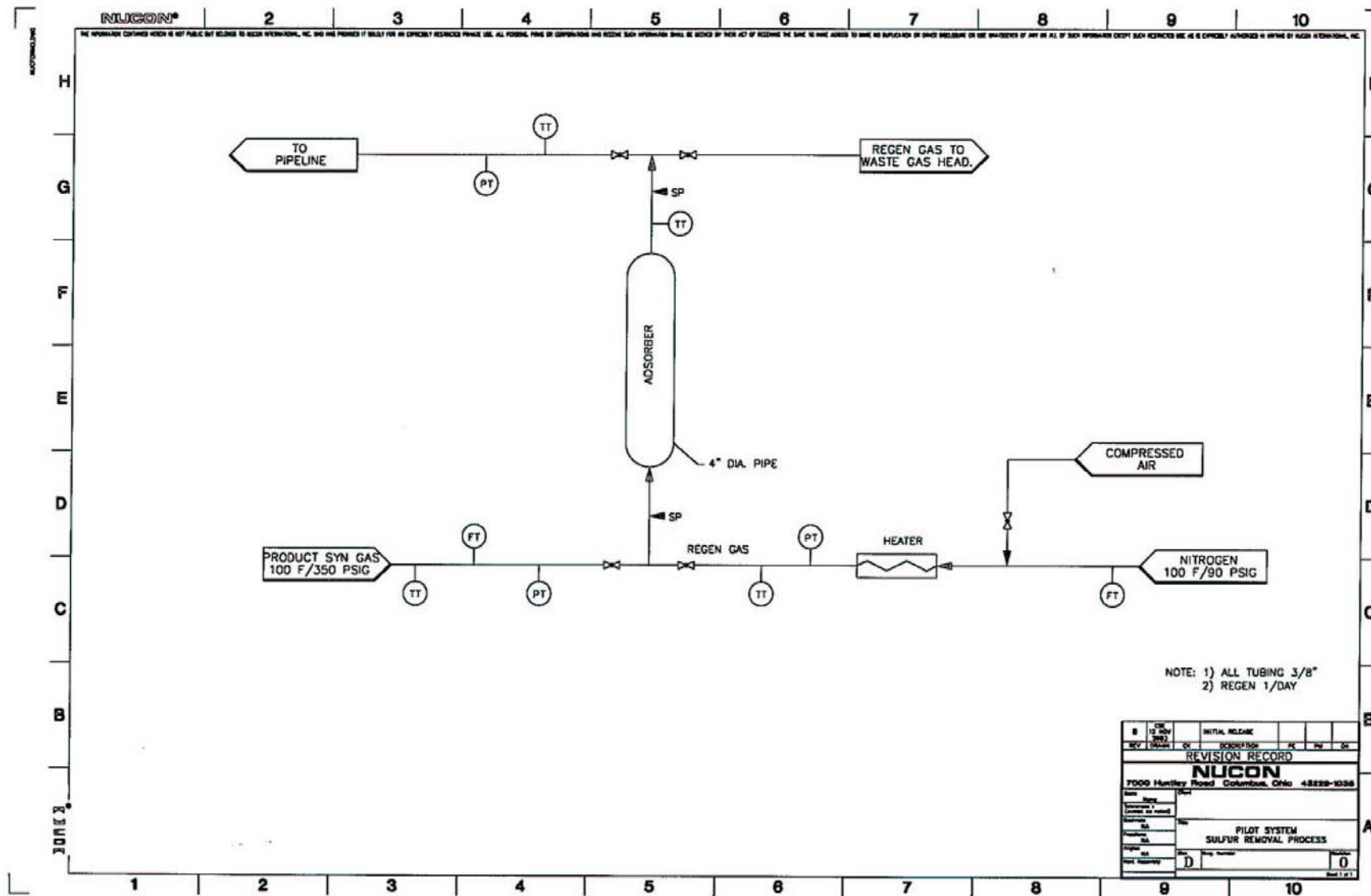


Figure 5.2.2.1 P&ID for Nucon Slipstream Unit

### 5.3 Direct Oxidation Sulfur Recovery (GTI/TDA)

TDA has completed the design and procurement of most of the equipment necessary to modify their existing direct oxidation apparatus for the slipstream test at the WREL plant and have begun construction of the unit. TDA is currently sizing some of the final components and completing the assembly of the apparatus.

#### 5.3.1 Equipment Modification

##### Description of the Apparatus

By the end of December 2003, TDA had completely designed the unit and are in the process of completing construction of the apparatus. Some of the components that needed to be purchased to make the modifications are listed in Table 5.3.1.1:

**Table 5.3.1.1 Select Equipment Cost for Retrofit**

Quantity	Item Description	Cost	Purchased?
2	Sentry dual heat transfer coils	\$1,600	Y
6	Re-ranged and calibrated MFCs	\$3,100	Y
5	TC probes	\$130	Y
3	High pressure brass cylinder regulators	\$600	Y
1	High pressure SS cylinder regulator	\$360	Y
1	Badger Meter research control valve	\$1,700	
1	Heated circulating bath	\$2,000	Y
2	Reactors (SCH 80 2.5" pipe & 300 lb. welded flanges	\$600	Y
1	Oxigraf laser diode O <sub>2</sub> analyzer	\$4,750	Y
20	Amptek heating tapes (various lengths)	\$1,000	Y
6	5-pack weld-on thermocouples	\$300	Y
6	Dual thermocouple probes	\$350	Y
Total:		\$17,690	

Prior to modifying the apparatus the existing direct oxidation (DO) apparatus at TDA had to be mostly disassembled because it was designed for low-pressure operation and used a Pyrex reactor. Obviously, Pyrex can only be used near atmospheric pressure and a new (stainless steel) high-pressure reactor had to be designed and fabricated. The syngas pressure at WREL is approximately 375 psig. A high-pressure reactor has been designed and is currently being fabricated. The reactor is essentially a long spool piece of 2.5 inch SCH80 pipe that uses 300 lb flanges at the top and bottom.

The apparatus has a feed section for admitting syngas into the system, a reactor section, a sulfur condenser system, and a gas analysis section. Hot syngas first passes through a computer controlled pneumatic shut off valve and then through an orifice plate for measuring the flow rate. Use of a coriolis flow meter for this application was considered but the cost was prohibitive (ca. \$7100). So a system to use an orifice plate with a spare

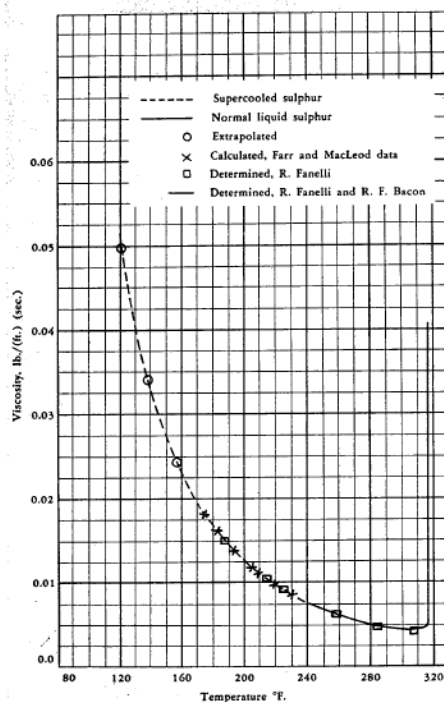
in parallel was designed and implemented. Based on the flowrate and the feed gas composition ( $\text{H}_2\text{S}$  concentration) obtained using gas chromatography, air will be metered into the system at the appropriate rate using an electronic mass flow controller so that the  $\text{H}_2\text{S}/\text{Oxygen (O}_2\text{)}$  ratio = 2. This corresponds to the stoichiometric amount of  $\text{O}_2$  required to oxidize the  $\text{H}_2\text{S}$  into sulfur and water. From there, the gas is either sampled through a reactor bypass (to measure the composition of the gas entering the catalyst bed) or the hot gas is directed into the catalytic reactor and over the catalyst bed.

The reactor is a fixed-bed design with a catalyst bed that has a volume of about  $300 \text{ cm}^3$ . The reactor will be made from 2.5 inch, SCH80 (heavy wall), 304 stainless steel pipe with 300 lb flanges welded to each end. Flange blanks will be drilled and tubing will be welded in place to accommodate the syngas inlet and outlet streams as well as tubing to permit inserting thermocouples into the reactor. The thermocouples will permit one to monitor the reactor temperature and determine the temperature profile through the reactor. The reactor body will be about 30 inches long and will be heated using a 3-zone tube furnace that is about 24-inches long (there are three, 6-inch heated zones).

Hot gas exiting the reactor contains unreacted syngas, elemental sulfur vapor, and water (as steam). The sulfur vapor will be condensed using two Parker heat transfer coils attached in series. These coils use a  $\frac{1}{2}$ -inch diameter stainless steel tubing inside of 1-inch diameter copper tubing that is wound into a coil (by the factory) to minimize size. Sulfur melts at approximately  $240^\circ\text{F}$  but if over heated (ca.  $370^\circ\text{F}$ ) sulfur will polymerize accompanied by a great increase in viscosity (Tuller 1954).

Figure 5.3.1.1 show the viscosity of liquid sulfur below about  $320^\circ\text{F}$  and Figure 5.3.1.2 shows the viscosity of liquid sulfur between  $300^\circ\text{F}$  and  $600^\circ\text{F}$ . Referring to both Figures, sulfur condensation has to be done within about  $90^\circ\text{F}$  of its melting point (ca.  $240^\circ\text{F}$ ). The condensers will be maintained at about  $260^\circ\text{F}$  using a hot-oil circulation bath.

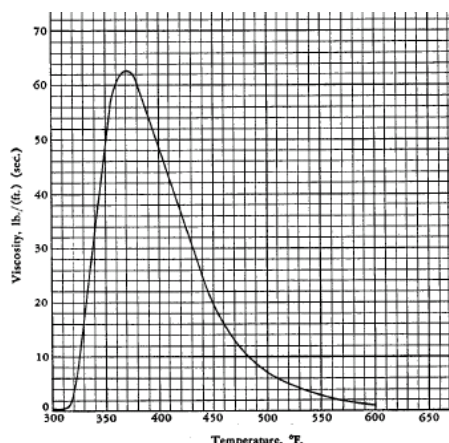
While it is impossible to keep the shell-side liquid completely isothermal, by operating the bath at a high flow rate, the temperature of the shell-side fluid can be kept relatively constant at about  $250\text{--}260^\circ\text{F}$ . Two heat transfer coils will be used in series to meet the heat transfer surface area requirements. Most of the heat duty is sensible cooling of the desulfurized syngas from  $400^\circ\text{F}$  to  $260^\circ\text{F}$ ; the amount of heat released by sulfur condensation is only 5% of the total heat load.



**Figure 5.3.1.1. Viscosity of liquid sulfur in low temperature range**

Liquid sulfur exiting the bottom coil flows into a 2-liter stainless steel pressure cylinder (Whitey sample cylinder available through Swagelok) through stainless steel tubing. This “sulfur collection pot” is kept at about 260°F to keep the sulfur molten. Band heaters are attached to the outside of the vessel, which is well insulated (as are all of the heated components of the apparatus) with mineral wool and fiberglass tape. A heat traced ball valve is located at the bottom of the 2-liter vessel to permit draining the liquid sulfur.

After the bulk of the sulfur vapor has been condensed, the syngas stream still contains sulfur vapor in a concentration of about 50-75 ppm which is approximately the equilibrium concentration of sulfur vapor over liquid sulfur at the condensation temperature of 260°F. Some sulfur aerosol particles might be entrained in the gas stream as well. When the temperature of the system drops, this additional sulfur will deposit in the system unless it is removed. For this reason, a



**Figure 5.3.1.2. Viscosity of liquid sulfur in high temperature range**

sulfur vapor knockout downstream of the sulfur condenser and collection vessel is installed. This is the horizontal cylinder in Figure 5.3.1.3 to the right of the condensers in the figure. The cylinder is a Whitey stainless steel sample cylinder rated for use at 1800 psig. It is packed with Pyrex wool and maintained at a temperature of about 100°F by means of band heaters. The temperature of 100°F is chosen because this temperature is below the dew point of sulfur vapor but is above the dew point of the water vapor in the syngas. Therefore, the sulfur (but very little water) should be trapped in this vessel. The glass wool provides a tortuous path to improve the collection efficiency of the vessel.

To be able to control pressures at 400 psig at flow rates of 5 – 10 actual liters/min, the control valve has to have a very small flow coefficient ( $C_v = 0.00008$ ). Consequently, any solid particles in the gas stream can easily cause the valve to jam or plug. Therefore, downstream of the horizontal sulfur vapor knockout are two inline filters. The first is a 15  $\mu\text{m}$  filter and the second is a 7  $\mu\text{m}$  filter. The filters are replaceable and are made from sintered 304 stainless steel (Swagelok). There is also a 0.5  $\mu\text{m}$  filter gasket just upstream of the pressure control valve (PCV). A heat traced bypass is plumbed in around the PCV so that the PCV can be serviced without necessitating a complete shutdown of the apparatus. The bypass or the PCV can be isolated using the shutoff valves.

Downstream of the control valve, the pressure is approximately ambient and the flow can be as high as 3 SCFM. The main flow passes to the vent/flare system through a back pressure regulator (BPR). The BPR permits sustaining a constant 1-5 psi back pressure on the system downstream of the PVC but upstream of the vent. This slight back pressure drives a low flow of gas (e.g. 500 mL/min) toward the gas chromatograph (GC) and through the O<sub>2</sub> analyzer. Since the GC sampling valve uses 1/16 inch tubing, most of

the flow goes around the GC and a much lower flow (ca 20-30 mL/min) is forced through the gas sampling valve using a needle valve (MV-10 in Figure 5.3.1.3).

#### Analytical Method

The GC is equipped with a thermal conductivity detector (TCD) for measuring the concentrations of H<sub>2</sub>, CO, methane (CH<sub>4</sub>) etc., plus higher concentrations of H<sub>2</sub>S and (if any) sulfur dioxide (SO<sub>2</sub>). A flame photometric detector (FPD) is used for measuring low concentrations of sulfur compounds. The concentration of hydrogen can be measured by TCD by using a carrier gas that contains 2% H<sub>2</sub> in helium (He). This carrier gas has a slightly higher thermal conductivity than pure helium because gas thermal conductivity depends mostly on molecular weight. With the slightly higher thermal conductivity carrier gas (2% H<sub>2</sub>/He), the negative then positive peakshape observed when H<sub>2</sub> elutes using pure He carrier gas is eliminated and the H<sub>2</sub> peak is completely above the base line (i.e. always positive) and can therefore be integrated.

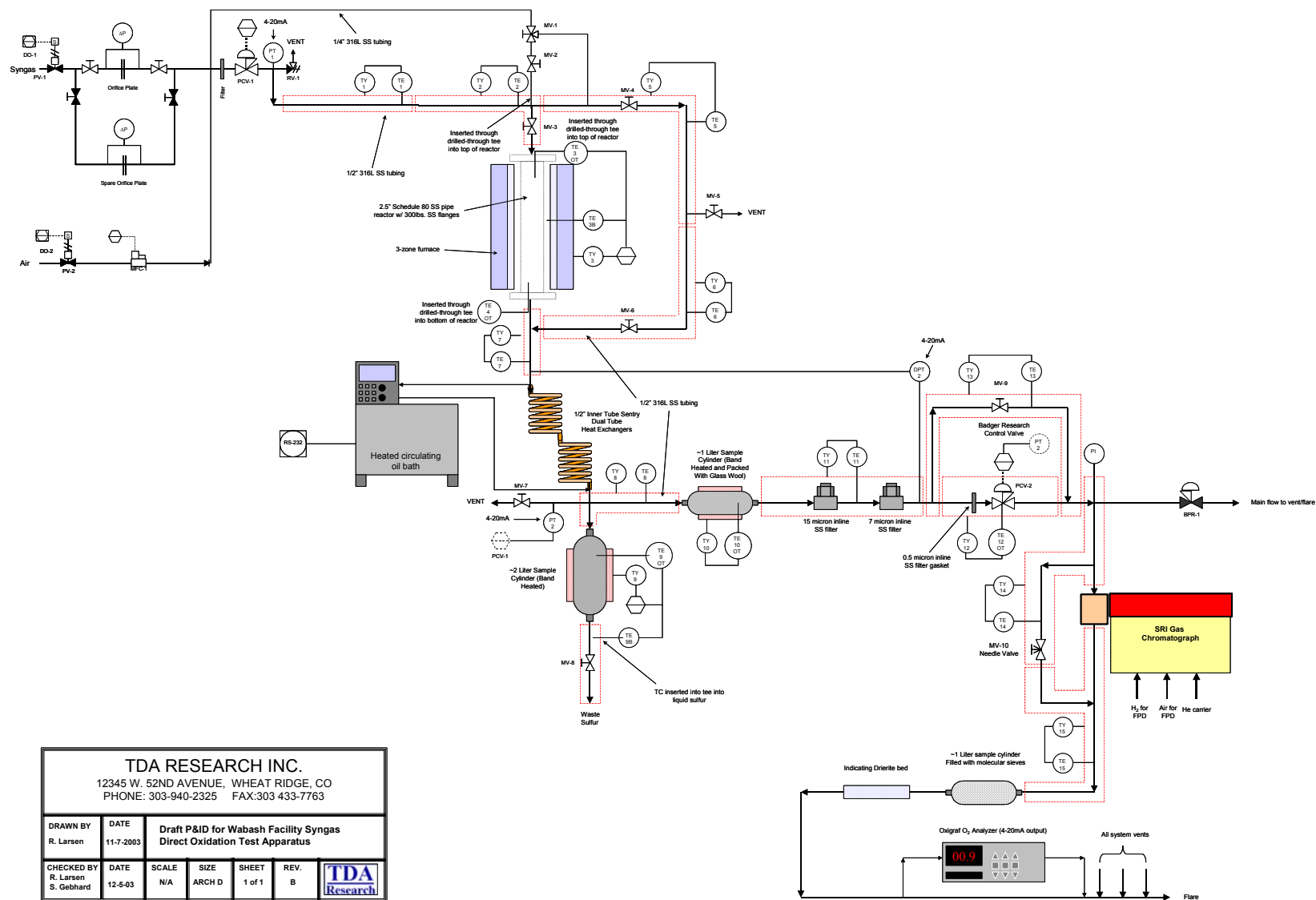
For measuring low concentrations of H<sub>2</sub>S, Carbon disulfide (CS<sub>2</sub>), COS, SO<sub>2</sub> and other sulfur compounds, the flame photometric detector (FPD) on the GC will be utilized. The FPD is similar to a flame ionization detector (FID) in that the sample is burned in a H<sub>2</sub> flame. The FPD is located downstream of the TCD because the FPD is a destructive analyzer whereas the TCD is not. Unlike an FID that measures an ion current when organic compounds are burned, the FPD measures the fluorescence that is emitted from excited states of gas phase diatomic sulfur (S<sub>2</sub>) species that are produced in a hydrogen/air flame. A bandpass filter is used in conjunction with a photomultiplier to measure the intensity of the correct wavelength (394 nm). This makes the FPD blind to anything but sulfur compounds. The FPD is also extremely sensitive with capability to easily detect H<sub>2</sub>S etc., in the 1-5 ppm range.

The gas that bypasses the GC passes through the O<sub>2</sub> analyzer in order to determine if any unreacted O<sub>2</sub> has passed through the catalyst bed (“O<sub>2</sub> slip”). The O<sub>2</sub> analyzer requires dry gas and therefore there is a molecular sieve bed followed by an indicating Drierite bed located just upstream of the O<sub>2</sub> analyzer. The molecular sieve removes the majority of the water from the gas being analyzed and the Drierite is used to signal water breakthrough (blue to pink color change). Gas exiting the O<sub>2</sub> analyzer is safely vented.

#### Design Details

For a detailed discussion of the design of the major components of the system, including the gas feed system, reactor, and sulfur condenser, please refer to Appendix 9.2.

Figure 5.3.1.3. P&ID for TDA Slipstream Unit



## 6.0 CONCLUSIONS

Phase II of the IMPPCCT Project was successfully launched during the reporting period. The objective of Phase II is to conduct research, development, and testing (RD&T) as outlined in the Phase I RD&T Plan to enhance the development and commercial acceptance of coproduction technology. Studies will address the technical concerns that will make the IMPPCCT concept competitive with natural gas-based systems in the commercial marketplace. Efforts in Phase II will investigate the cleanup of the syngas by removing contaminants, particularly sulfur species, to a level acceptable for the methanol synthesis catalyst, and reducing the cost of the current sulfur removal system such as via warm gas cleanup methods. Potential technologies being evaluated include Nucon's regenerable activated carbon and TDA's direct sulfur oxidation process to remove hydrogen sulfide and other sulfur species. Laboratory testing followed by on-site testing at WREL with bench-scale slipstream units will be conducted. Sulfur removal from actual syngas produced by the facility will be evaluated at system pressure and temperature.

### 6.1 Project Management

Both Nucon and GTI, subcontractors of COP, were formally notified to start on their project under the IMPPCCT Phase II contract awarded COP by DOE, while a subcontract document with COP was being executed. GTI has in turn notified its subcontractor, TDA, of the award. Towards the end of the reporting period, draft subcontract agreements that include language on non-disclosure were sent to Nucon and GTI for review and comment.

Project kickoff meetings were held at TDA and Nucon. Team members were apprised of the background and objectives of the IMPPCCT project, and were aligned on the objectives, schedule, milestones, and deliverables, as well as invoicing procedures, on the project.

To ensure the RD&T is executed in an orderly and organized manner, COP prepared a Phase II RD&T Project Management Plan (PMP) with input from the subcontractors. The draft PMP was submitted to the DOE Project Manager for review at the end of the reporting period.

### 6.2 Regenerable Carbon for Sulfur Polishing (by Nucon International)

#### *6.2.1 Conduct Laboratory Tests to Determine the Optimum Operating Condition for Slipstream Testing*

Initial experiments estimating the proper parameters of column diameter and length have been performed. The initial experiments used a gas mixture containing 11% carbon dioxide, which interfered with COS measurements. Additionally, the content of COS and H<sub>2</sub>S were in excess of 100 ppm each. Following this, a short series of experiments with impregnated carbon were performed using a gas mixture containing 50 ppm H<sub>2</sub>S in nitrogen. These experiments established that for the laboratory testing, a 2-inch bed depth was appropriate (0.296 in ID) with the 20 x 40 mesh activated carbon impregnated with the metal oxides. Upon receiving the initial syngas mixture (Praxair), each of the singly impregnated carbon preparations was evaluated for an initial assessment of breakthrough and times to saturation. Capacity for H<sub>2</sub>S and COS for

each impregnated carbon was determined. Good capacities were obtained for all impregnated carbons.

#### ***6.2.2 Design, Procure and Fabricate Slipstream Unit.***

Nucon has prepared a preliminary process design description and P&ID. These will be further refined.

#### **6.3 Direct Oxidation Sulfur Recovery (by GTI/TDA)**

TDA has completed the design and procurement of most of the equipment necessary to modify their existing direct oxidation apparatus for the slipstream test at the WREL plant and have begun construction of the unit. TDA is currently sizing some of the final components and completing the assembly of the apparatus.



## 7.0 MILESTONES & PLANS

### 7.1 Project Schedule and Milestones

Figure 7.1.1 is the IMPPCCT Project Phase II Schedule as represented in the Project Management Plan (PMP). The project has a late start due to the transitioning activities associated with the acquisition of GEC and E-Gas by COP from Global Energy in late summer of 2003. It is optimistic that the project will be able to pick up speed after the successful launch of the Nucon and TDA projects during the reporting period to get back on the original schedule.

The project successfully completed its first milestone during the reporting period, which is to “Initiate construction of bench-scale, field-test units to remove sulfur and other trace contaminants present in synthesis gas”.

### 7.2 Plan for Next Reporting Period

#### 7.2.1 Project Management

COP plans to complete executing the subcontracts with Nucon and GTI, and GTI will complete their subcontract with TDA. Revisions and comments from DOE on the PMP will be incorporated and a final PMP will be issued.

#### 7.2.2 Regenerable Carbon for Sulfur Polishing (by Nucon International)

##### Conduct laboratory tests

As a result of discussions with COP, a new syngas mixture has been ordered with levels of H<sub>2</sub>S and COS more closely matching the expected conditions that the client is anticipating. The following tests will be performed:

1. Testing of each single impregnant with the higher H<sub>2</sub>S concentration (300 ppm)
2. Testing each single impregnant with COS alone (25 ppm) to evaluate the conversion of COS to H<sub>2</sub>S and to more accurately determine the behaviour of each carbon treatment toward COS
3. Testing of metal combinations in the carbon impregnation (still 8% total metal oxide content)
4. Scaling up the column diameter to evaluate the performance of pelleted carbons (1.5 and 3.0 mm)
5. Generating select data on the regeneration of the metal oxide impregnated carbons that show the most promise for this application.

### Design, Procure and Fabricate Slipstream Unit

A process safety review will be conducted when the design is agreed upon. The primary metal components (4-inch diameter pipe and 3/8-inch tubing) are readily available. Some instrumentation and control components are available from NUCON stocks and others will be acquired. It is anticipated that the slipstream unit can be completed on schedule by the end of March 2004.

#### ***7.2.3 Direct Oxidation Sulfur Recovery (by GTI/TDA)***

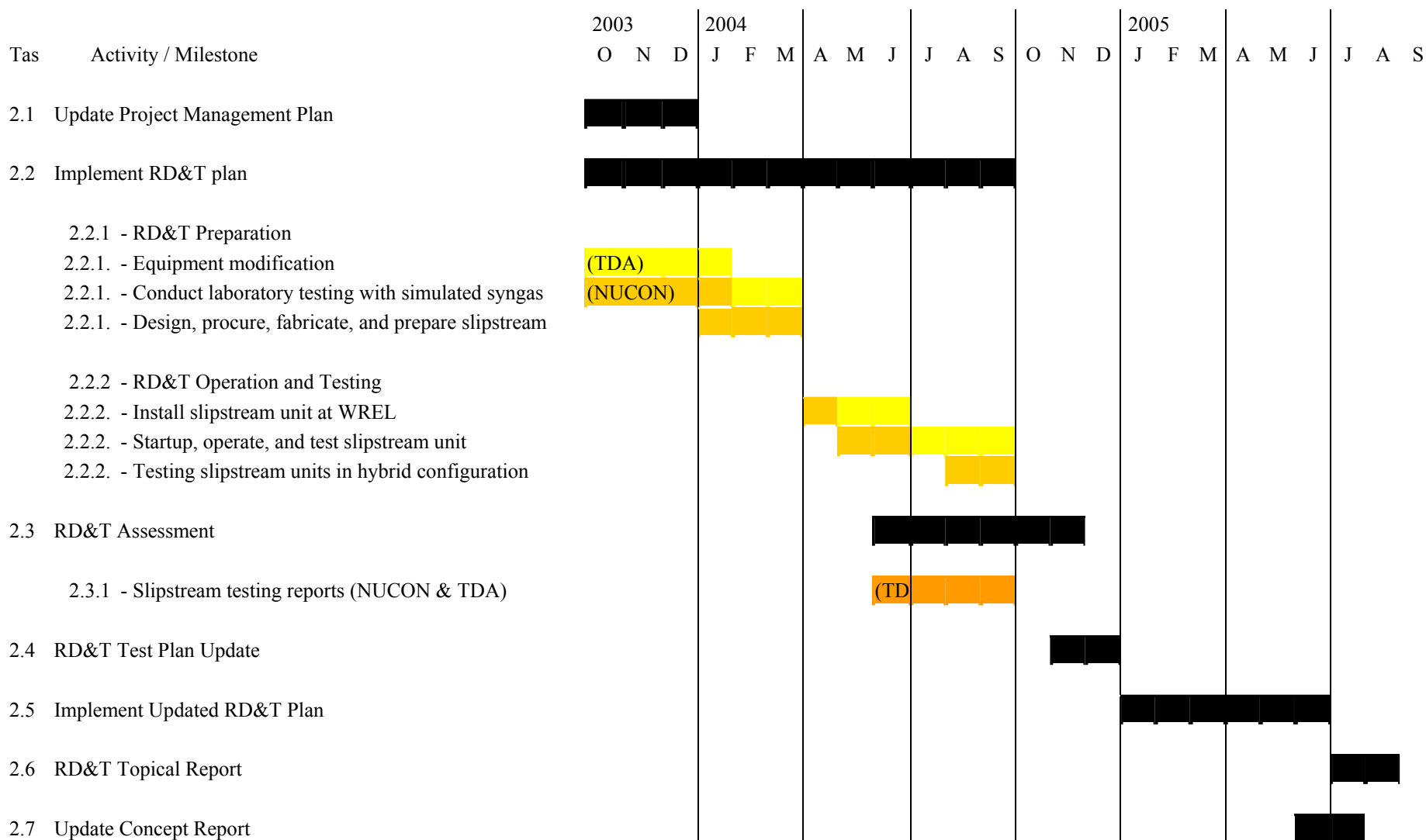
TDA will continue to retrofit and modify the existing direct oxidation apparatus for the desulfurization slipstream test at WREL. The modification should be complete and the unit ready for laboratory testing by the end of the reporting period.

### **7.3 Project Spending**

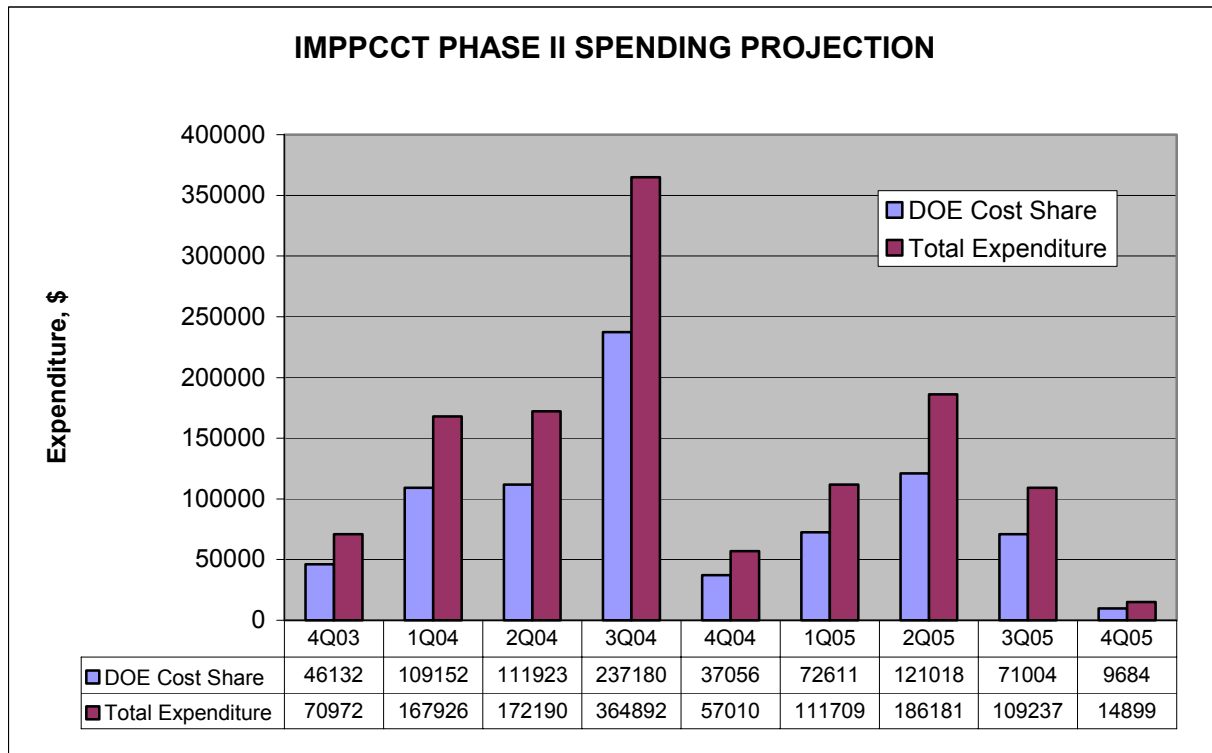
For the reporting period, Nucon has submitted an invoice for \$8,054.09. GTI has not submitted an invoice due to the delay in finalizing and executing the subcontract with COP. An invoice is being prepared by COP for submission to DOE via Global Energy. Invoices will be submitted to DOE directly once the project has been novated from Global Energy to COP.

The projected spending schedule as submitted in the PMP is shown in Figures 7.3.1 and 7.3.2. Once GTI's invoice is received, total expenditure for the reporting period will be compared to the projected figures.

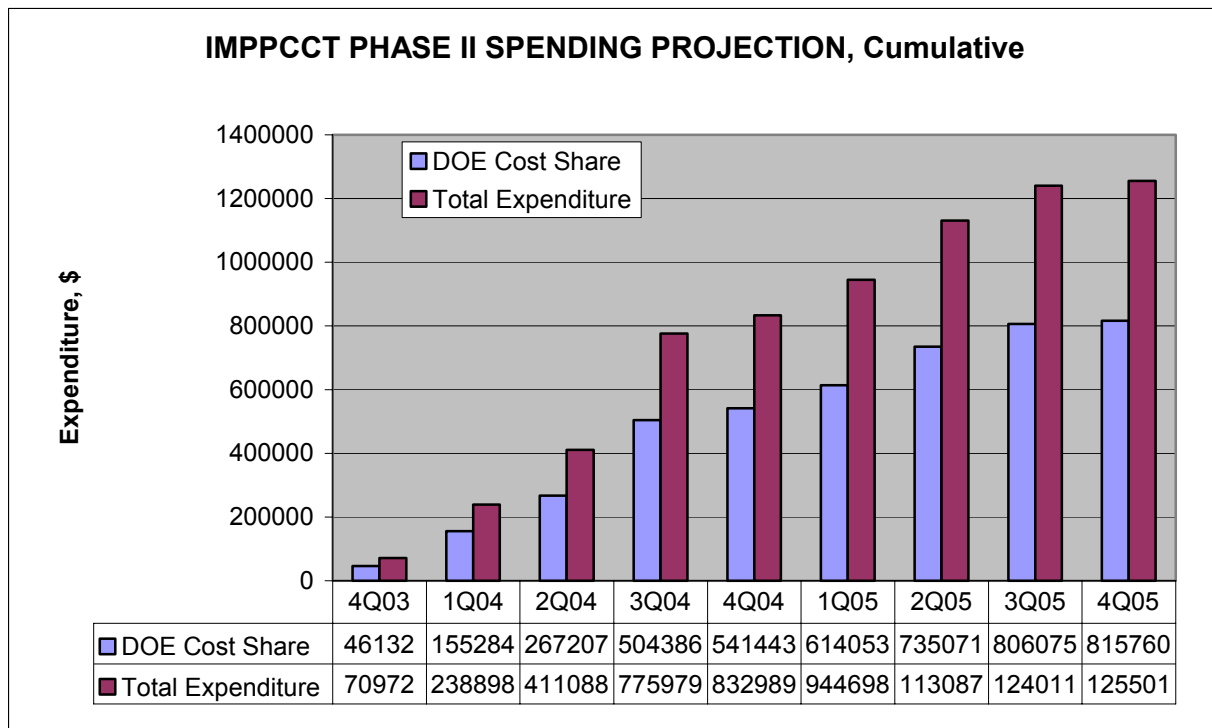
**Figure 7.1.1 : IMPPCCT Phase II Project Schedule**



**Figure 7.3.1: Phase II Spending Projection**



**Figure 7.3.2: Phase II Spending Projection -- Cumulative**



## 8.0 REFERENCES

### 8.1 Selected References on WREL and E-Gas Available via the Internet

1. **“National Energy Policy”, issued by the White House in May 2001.** The Wabash River facility and Global Energy received mention in a dedicated sidebar on page 3-6.  
<http://www.whitehouse.gov/energy/Chapter3.pdf>
2. **“Wabash River Coal Gasification Repowering Project, An Update”,** Department of Energy Topical Report No. 20, September 2000, summarizes the history of the Wabash River facility and its construction and four year demonstration under the DOE’s Clean Coal Technology program.  
<http://www.lanl.gov/projects/cctc/topicalreports/documents/topical20.pdf>
3. **“Wabash River Coal Gasification Repowering Project Final Technical Report”,** August 2000, 358 pages. This is a very detailed look at the Wabash River facility and its operation 1995-1999.  
[http://www.lanl.gov/projects/cctc/resources/pdfs/wabsh/Final%20\\_Report.pdf](http://www.lanl.gov/projects/cctc/resources/pdfs/wabsh/Final%20_Report.pdf)
4. **“Gasification Plant Performance and Cost Optimization”,** May 2002, (23 MB). The final report of Task 1 of this comprehensive (\$2.4 million) study performed by Global Energy, Nexant and Bechtel under subcontract to the DOE to identify cost savings in the next generation of integrated gasification and coproduction facilities utilizing the E-Gas Technology. Detailed cost estimating by Bechtel.  
<http://www.netl.doe.gov/coalpower/gasification/projects/systems/docs/40342R01.PDF>
5. **“Wabash River Coal Gasification Repowering Project, A DOE Assessment”,** January 2002. This is the DOE’s official post-project assessment of the Wabash River project.  
<http://www.lanl.gov/projects/cctc/resources/pdfs/wabsh/netl1164.pdf>
6. **“Environmental Benefits of Clean Coal Technologies”** Department of Energy Topical Report No. 18, April 2001. This report describes a variety of processes that are capable of meeting existing and emerging environmental regulations and competing economically in a deregulated electric power marketplace  
<http://www.lanl.gov/projects/cctc/topicalreports/documents/topical18.pdf>
7. **“Coproduction of Power, Fuel, and Chemicals”** Department of Energy Topical Report No. 21, September 2001. A description of the production of synthesis gas (syngas) from coal, the production of electricity from combusting a portion of the syngas and conversion of the remaining syngas to high-value fuels and chemicals.  
<http://www.lanl.gov/projects/cctc/topicalreports/documents/topical21.pdf>
8. **The Gasification Technology Council** maintains a website ([www.gasification.org](http://www.gasification.org)) that includes a library of the papers presented at recent conferences. Papers presented by Global Energy in 2002 & 2001:
  - “Wabash River Repowering IGCC Operations and Performance Update Report”, October 2002  
[http://www.gasification.org/Presentations/2002\\_papers/GTC02010.pdf](http://www.gasification.org/Presentations/2002_papers/GTC02010.pdf)
  - “Comparative IGCC Cost & performance for Domestic Coals”, October 2002  
[http://www.gasification.org/Presentations/2002\\_papers/GTC02018.pdf](http://www.gasification.org/Presentations/2002_papers/GTC02018.pdf)
  - “NOx Control in IGCC Combustion Turbines: Steam vs. Nitrogen”, October 2002  
[http://www.gasification.org/Presentations/2002\\_papers/GTC02022.pdf](http://www.gasification.org/Presentations/2002_papers/GTC02022.pdf)
  - “Optimized Petroleum Coke IGCC Coproduction Plant”, October 2001  
<http://www.gasification.org/98GTC/GTC01018.pdf>
  - “Environmental Performance of IGCC Repowering for Conventional Coal Power Plants”, October 2001  
<http://www.gasification.org/98GTC/GTC01037.pdf>

## 8.2 Other References

Baumeister, T.; Avallone, E.A. and Baumeister III, T. (1978) Mark's Standard Handbook for Mechanical Engineers, 8<sup>th</sup> ed., McGraw Hill, p 8-182.

Higdon, A.; Ohlsen, E.H.; Stiles, W.B.; Wesse, J.A. and Riley, W.F. (1976) Mechanics of Materials, 3<sup>rd</sup> ed., Wiley, pp. 157-166.

Perry, R.H., Green, D.W., and Maloney, J.O. (1999) Perry's Chemical Engineers' Handbook, 7<sup>th</sup> ed., McGraw Hill, Ch 10.

Shigley, J.E. (1977) Mechanical Engineering Design, 3<sup>rd</sup>, ed. McGraw Hill, pp. 170-173.

Tuller, W.N. (1954) The Sulfur Data Book, Freeport Sulfur Corporation. p. 22

## 9.0 APPENDIX

### 9.1 TDA Slipstream Unit Design Details

This section discusses the design of the major components of the system that are not standard items or that have to be properly sized using appropriate calculations.

#### *Gas Feed System*

The apparatus was designed to operate at a flow rate of 5 liters/min at approximately 400 psig and 400°F. The mechanical calculations (discussed later) were done for a pressure of 500 psi. A gas composition (COP proprietary) from the WREL gasifier was used to calculate transport properties of the syngas (density, viscosity, heat capacity, etc.). Only the major components were included in the calculations and the transport properties were calculated using the NIST computer program, Supertrapp.

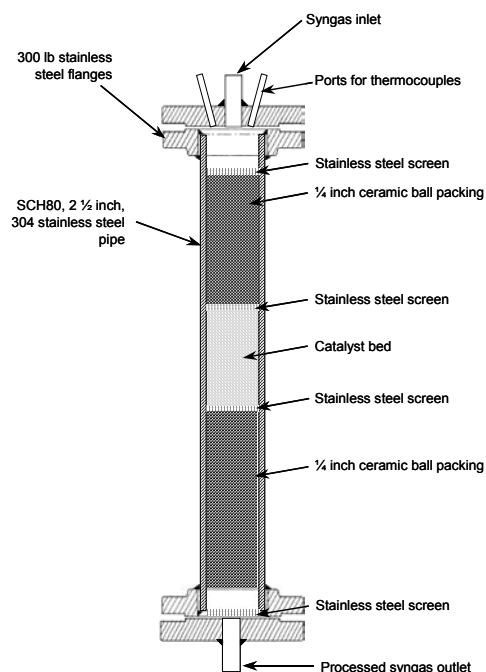
Properties such as viscosity and thermal conductivity are needed for calculating the Reynolds and Prandtl numbers for the heat transfer calculations used to size the sulfur condensers. Because the sulfur loading is fairly low, it turns out that 95% of the heat duty of the sulfur condensers is in cooling the gas from reaction temperature (ca. 400°F) down to 260°F.

The fluid properties calculated from Supertrapp will be used to calculate the gas flow rate from the orifice plate pressure drop. There will be two orifice plates in parallel to permit continuous operation in the event that one of the plates plugs up and needs to be serviced.

Just downstream of the orifice plate assemblies, the gas is either directed to bypass the reactor for inlet gas analysis, or is mixed with O<sub>2</sub> (as air) and sent to the reactor. The amount of O<sub>2</sub> (as air) added is determined from the inlet gas analysis and the feed flow rate; only enough air is added to provide enough O<sub>2</sub> to oxidize the H<sub>2</sub>S into sulfur and water. At the correct air flow, H<sub>2</sub>S/O<sub>2</sub> ratio = 2 for the reaction  $\text{H}_2\text{S} + 0.5\text{O}_2 \rightarrow \text{S} + \text{H}_2\text{O}$ . Air is metered into the system using an electronic mass flow controller. At a flow rate of 5 actual liters/min and an H<sub>2</sub>S concentration of 1.5%, the unit will produce approximately 5 lb/day of elemental sulfur (assuming 90% H<sub>2</sub>S conversion).

#### *Catalytic Reactor*

The reactor (Figure 9.1.1) is a fixed bed design with a catalyst bed volume of about 300 cm<sup>3</sup>. Inert ceramic balls (1/4 inch) are placed above and below the catalyst giving a total bed volume of over 500 cm<sup>3</sup>. The reactor will be made from 2.5-inch, SCH80, 304 stainless steel pipe with 300 lb flanges welded to each end. Flange blanks will be drilled and tubing will be welded in place to accommodate the syngas inlet and outlet as well as tubing to permit inserting thermocouples into the catalyst bed and into the ceramic ball beds. The thermocouples



**Figure 9.1.1 Simplified schematic of high-pressure fixed bed catalyst test**

will be used to monitor the reactor temperature and determine the temperature profile through the bed. The reactor will be about 30 inches long and will be heated in a three zone tube furnace that is about 24-inches long (there are three 6 inch heated zones).

#### *Catalyst Bed*

The catalyst bed volume is about 500 cm<sup>3</sup> based on an actual flowrate of 5 liters/min at 390°F and 375 psig and a space velocity of 1000 cm<sup>3</sup><sub>gas</sub>/cm<sup>3</sup><sub>catalyst</sub>/hour. To support the bed and act as a gas diffuser, the reactor above and below the catalyst bed will be packed with ¼ inch ceramic balls. The catalyst is separated from the ball-bed portions using stainless steel screens. The residence time of the gas in the catalyst bed will be about 3-5 seconds depending on the exact syngas flow rate. The pressure drop through the bed is expected to be about 0.5 inches of water column at 5 liters/min actual syngas flowrate.

#### *Stress Analysis of the Reactor Body*

The reaction will be made from 2.5-inch SCH80, 304L SS pipe so that the catalyst in the form of 1/8 x ¼ inch pellets can be used. This is the actual form of the catalyst that would be used in a full-scale application. By using the catalyst in the same form, the transport effects during the slip-stream test will be closer to those that will occur in the larger unit.

To determine if the wall thickness and other mechanical properties of the reactor would be satisfactory, a stress analysis was performed for the reactor (and all of the other components as well) and calculated the various safety factors for high pressure operation. The equations used to calculate the radial (Equation 1), axial (Equation 2), and tangential stresses (Equation 3) in the reactor are shown below. The calculations were done assuming a 500 psi internal pressure. Figure 9.1.2 shows the definitions of the various terms given in the equations (Higdon et al., 1976). Because the reactor has flanges on the end, there is an axial force that tries to stretch the pipe body because of the pressure inside the reactor pushing on the ends of the cylindrical volume. Therefore, a simple “maximum hoop stress” calculation may be insufficient to calculate the safety factor for the reactor. The axial stress in the reactor due to the forces on the ends was calculated to be approximately 500 psi.

$$\sigma_{\text{radial}} = \frac{a^2 P_{\text{internal}} - b^2 P_{\text{external}}}{b^2 - a^2} - \frac{a^2 b^2 (P_{\text{internal}} - P_{\text{external}})}{(b^2 - a^2) p^2}$$

**Equation 1. Radial stress.**

$$\sigma_{\text{axial}} = \frac{F_{\text{axial}}}{A_{\text{metal}}} + (P_{\text{internal}} - P_{\text{external}})$$

**Equation 2. Axial stress.**



$$\sigma_{\text{tangential}} = \frac{a^2 P_{\text{internal}} - b^2 P_{\text{external}}}{b^2 - a^2} + \frac{a^2 b^2 (P_{\text{internal}} - P_{\text{external}})}{(b^2 - a^2) \rho^2}$$

Equation 3. Tangential stress

The radial stress is the stress in the radial direction and this was also about 500 psi compressive (the expansion of the inner wall induces net compression in a thick walled cylinder). The highest stress in a cylinder under internal pressure is, as expected, tangentially oriented and occurs at the inner cylinder wall. This is the so-called hoop stress and is approximately 2400 psi in this case. If the cylinder did not have an axial load due to the pressure on the ends of flanges, then keeping the hoop stress below some design stress would be sufficient to calculate a safety factor. When an axial stresses are present, a distortion energy theory gives a more conservative design criterion (Shigley 1977). To apply this theory to design requires calculating the three principal stresses (zero shear stresses) in the system.

Using distortion energy theory in design, one calculates an equivalent stress in terms of the principal stresses, and then compares this equivalent stress to the allowable stress chosen for the design. The equation used to calculate the equivalent stress is shown in Equation. Fortunately with a cylinder, the axial, radial and tangential stresses are the principal stresses which simplifies the analysis; these stresses are included in Equation. In our case, the equivalent stress ( $S_y$ ) is about 2600 psi. Note that this is slightly higher than the tangential stress alone (2400 psi). In this case, the difference in stresses happens to be minor, but for pressure vessels with large axial loads, the difference can be significant, enough to require lower internal pressures or thicker walled vessels.

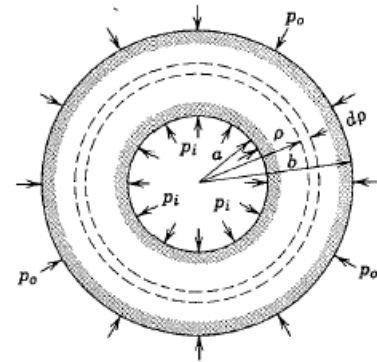


Figure 9.1.2 Thick walled cylinder definitions.

$$S_y = \left[ (\sigma_{\text{radial}} - \sigma_{\text{axial}})^2 + (\sigma_{\text{axial}} - \sigma_{\text{tangential}})^2 + (\sigma_{\text{tangential}} - \sigma_{\text{radial}})^2 \right]^{\frac{1}{2}}$$

Equation 4. Equivalent stress from principle stresses in the cylindrical reactor under internal pressure.

The maximum allowable stress used for designing the reactor was 0.2% offset, which for 304 stainless steel is 23,000 psi at 401°F as shown in Table 9.1.1. Comparing the 23,000 psi allowable stress with 2600 psi equivalent stress in the reactor gives an operational safety factor of about 9 using 5.

$$SF \equiv \frac{\sigma_{\text{allowable}}}{S_y}$$

Equation 5. Definition of the Safety Factor

Temp(F)	0.2% offset
70	35,000
401	23,000
801	19,000
1202	15,500
1499	13,000
1832	7,250

Table 9.1.1 304 stainless steel strength data.

### *Reactor Flanges*

The reactor will use 300 lb flanges at each end. The 300 lb designation refers to the pressure rating for continuous operation at 850°F (Table 9.1.2). At 400°F, the normal operating temperature of the reactor, the flanges are rated for continuous service at a pressure of 665 psi which is well above the 375 psig normal operating pressure. Stainless steel 316 flanges will be used. The flanges will be drilled out for weld fittings to accommodate inlet and outlet tubing, thermocouples, pressure taps, etc.

	"150 lb"	"300 lb"
(°F)	Pressure	Pressure
100	275	720
200	240	700
300	210	680
400	180	665
500	150	625
600	130	555
700	110	470
800	92	365
850	82	300
900	70	225
1000	40	85

### *Sulfur Condenser Heat Transfer.*

The total heat load for cooling the gas from 400°F to 260°F is about 550 Btu/hr. About 95% of the heat load is due to sensible cooling of the gas and 5% of the heat load is from the

heat of condensation of the sulfur. The Reynolds number (Re) of the gas in the condenser is about 6400.

**Table 9.1.2 Primary pressure rating for ANSI flanges (Baumeister et al.**

Unfortunately, this is in the transition region of flow; however, if the tubing I.D. is reduced to increase Re to turbulent levels, then the risk of plugging would be greatly increased. To be conservative (and over-designing the sulfur condenser) laminar flow correlations to calculate the heat transfer coefficient was used. This gave a heat transfer coefficient that was somewhat low but makes the heat exchanger calculations conservative. For laminar flow under these conditions the Nusselt number is  $Nu = 4.36$  which gives an inner tube heat transfer coefficient of about 4.5 Btu/ft<sup>2</sup>/hr/°F.

The gas is cooled by circulating a synthetic heat transfer oil through the "shell side" of the condenser. A circulation rate of 18 liter/min was chosen to keep the oil as isothermal as possible. At this flow rate the Reynolds number for the oil is 60,000 and turbulent flow correlations can be used. In this case, the heat transfer coefficient was approximately 175 BTU/ft<sup>2</sup>/hr/°F. When the thermal conductivity of the 316 stainless steel inner tube is considered ( $k \approx .9$  BTU/ft/hr/°F) the overall heat transfer coefficient is only 4.3 Btu/ft<sup>2</sup>/hr/°F (as usual, the gas side heat transfer coefficient controls the rate of heat transfer).

For a gas inlet temperature of 400°F, an outlet temperature of 260°F, and liquid circulating at 250°F the log mean temperature difference is about  $LMTD = 52^\circ\text{F}$ . Thus, 2.4 ft<sup>2</sup> of heat transfer area is required to cool the gas to 260°F. One coil (surface area = 1.93 ft<sup>2</sup>) is inadequate but two coils gives an excess capacity of about 60%. Two coils were used in the design.

### Sulfur Condenser

The sulfur condensers are heat transfer coils (formerly made by Parker). These are basically simple tube-in-tube condensers. The inner tube is ½-inch O.D. 316 stainless steel and the outer tube is 1-inch O.D. copper. The exchangers meet the ASTM A-269 boiler code specifications. Figure 9.1.3 is a drawing of the heat transfer coil.

### Sulfur Condenser Stress Analysis

The same type of mechanical analysis used for the reactor (Section 0) was applied to all of the heated sections of the apparatus including the tubing and the sulfur condenser coils. The inner tube of the sulfur condenser is stainless steel and the jacket is copper. For the copper outer tube we used a maximum allowable stress of  $\sigma_{Cu} = 10,000$  psi. The resulting safety factor for the copper was 3.5 (assuming 500 psi internal pressure in case of a leak in the inner tube). The safety factor for the inner 316 SS tube was about 12 (again assuming  $P = 500$  psi and  $T = 260^{\circ}\text{F}$ ).

For standard items such as the pressure control valve, the stainless steel cylinders, tubing, etc., safety factors were calculated by using the manufacturers (Swagelok) continuous service pressures degraded for elevated temperature performance, which are shown in Table 9.1.3. In all cases, the safety factors were in the range of 3-5.

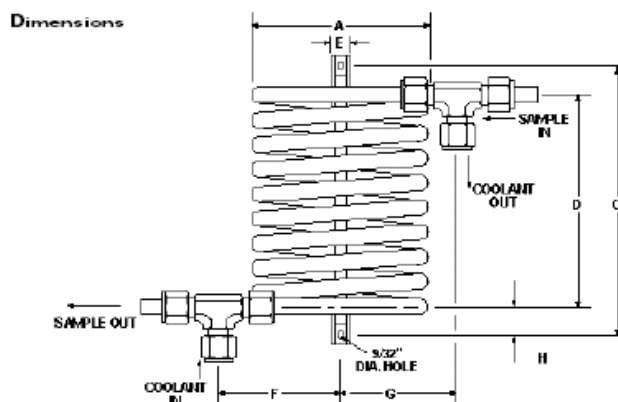


Figure 9.1.3 Parker heat transfer coil.

Factors							
°F	°C	Aluminum	Copper	Steel <sup>2</sup>	304SS	316SS	Alloy 400
200	93	1.00	0.80	0.95	1.00	1.00	0.87
400	204	0.40	0.50	0.87	0.93	0.96	0.79
600	315	—	—	—	0.82	0.85	0.79
800	426	—	—	—	0.76	0.79	0.75
1000	537	—	—	—	0.69	0.76	—
1200	648	—	—	—	0.30	0.37	—

Table 9.1.3 Swagelok Pressure Rating Factor

### Orifice Plate

TDA completed the design of a flow measurement and control apparatus utilizing orifice plates. In 1-inch diameter 316 SS inlet pipe, a circular, sharp edged orifice that is approximately 3-mm in diameter can be used to obtain a pressure drop that can be easily and accurately measured, and that is not so high that it would degrade the inlet pressure to the system to an unacceptable level (e.g. a 100 psi pressure drop would be unacceptable because the maximum inlet pressure to the apparatus would be reduced to 300 psig). Acceptable pressure drops are in the

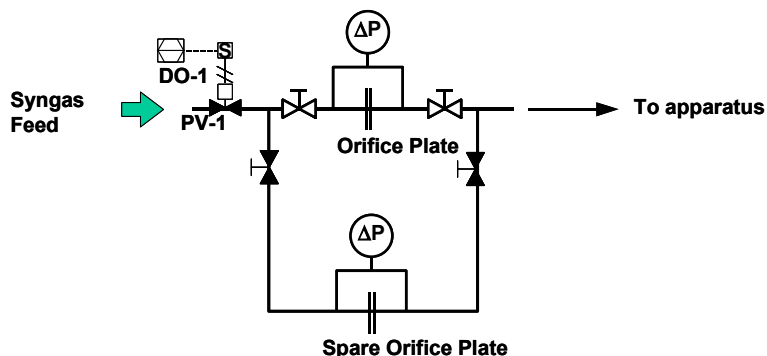


Figure 9.1.4 Parallel orifice plate configuration.

range of  $\Delta P = 5\text{--}10$  psi. If  $\frac{1}{2}$ -inch tubing were used, the required orifice would be 1.8 mm in diameter to achieve similar performance. Details of the calculations are outlined in the literature (Perry et al. 1999).

The system will use a pair of orifice plates so that one can be cleaned without having to take the apparatus out of service. The configuration is shown in Figure 9.1.4. A differential pressure reading will be recorded using a Validyne differential pressure transducer. The signal from the pressure transducer will be used by the process control computer to control the inlet syngas flowrate using the Badger Meter Co. control valve.

## **9.2 Project Kickoff Meeting Presentation by ConocoPhillips**

(Attached)

**Slipstream Testing of TDA's Direct Oxidation Sulfur Recovery Process at Wabash River IGCC Facility**

**Project Kick-off Meeting**

Albert Tsang, ConocoPhillips  
Raj Palla, Cbas Technology Institute  
Girish Srinivas, TDA Research

@ TDA Research, Wheat Ridge, CO

Oct 23, 2003

ConocoPhillips

Oct 23, 2003

ConocoPhillips

- ## AGENDA
- Background on IMPPCCT project (Albert)
  - Overview of EGas, Wabash Plant (Albert)
  - Overview of GTI and capability (Raj)
  - Overview of TDA and capability (Girish)
  - DOSR project overview, available data (Girish)
  - Discussion on Plan, Budget, Invoicing, Deliverables, Concerns, etc. (All)
  - Agreement on Plan (All)
  - Tour of TDA facility, see lab set up for DOSR
  - (mtg adjourned)

**Wabash River Integrated Methanol and Power Production from Clean Coal Technologies (IMPPCCT)**

Cooperative Agreement No. DE-FC26-99FT40659

**An Early Entrant Co-Production (EECP) Project**

\*\*\*\*\*

DOSR Project under:

- Task 2.2.1 – RD&T Preparation
- Task 2.2.2 – RD&T Operation
- Task 2.3 – RD&T Assessment

Oct 23, 2003

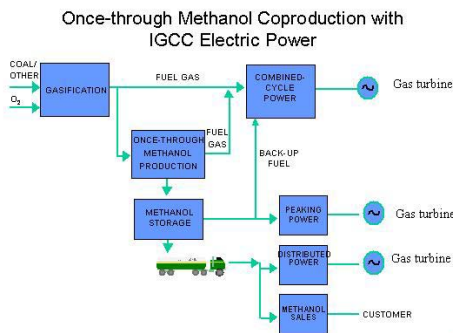
ConocoPhillips

Oct 23, 2003

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## IMPPCCT Objectives:

Development of an engineering design and economics for a single plant that produces methanol and electric power from the gasification of coal and other carbonaceous feedstocks

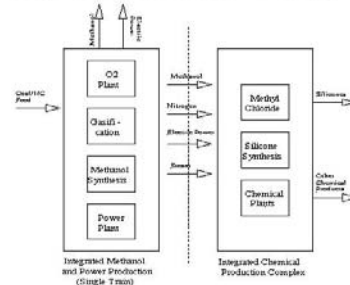


Oct 23, 2003

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## Integrated Chemicals Complex



Oct 23, 2003

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## Funding

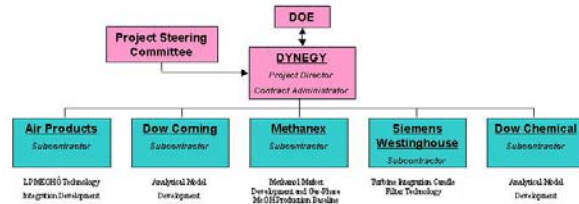
Budget Period	DOE Cost	Dynegy Power Corp.	Total	DOE Share	Recipient Share
1	\$ 1,546,902	\$ 386,726	\$ 1,933,628	80%	20%
2	\$ 1,003,810	\$ 540,513	\$ 1,544,323	65%	35%
3	\$ 721,052	\$ 721,052	\$ 1,443,004	50%	50%
Total	\$ 3,271,764	\$ 1,648,291	\$ 4,920,055		

Oct. 23, 2003

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## IMPPCCT Phase I Organization Chart



Oct. 23, 2003

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## Objectives for Phases I:

- Analyze and Develop a Concept for Methanol Production
- Complete a Feasibility Study for Wabash
- Evaluate Commercial Embodiment Plants
- Analyze the Domestic Methanol Markets
- Evaluate the economics

Oct. 23, 2003

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## Objectives for Phases II:

To Conduct RD&T to:

- Enhance the development and commercial acceptance of co-production technology
- Resolve critical knowledge and technology gaps on the integration of gasification and downstream processing

Oct. 23, 2003

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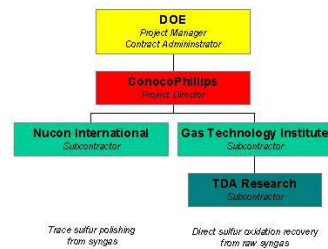
## Phase II Projects

- Slipstream Testing of Regenerable Activated Carbon to Remove Trace Sulfur Contaminants*  
(Nucon International)
- Slipstream Testing of TDA's Direct Oxidation Sulfur Recovery Process at Wabash River IGCC Facility*  
(TDA Research)

Oct. 23, 2003

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## IMPPCCT Phase II Organization Chart



Oct. 23, 2003

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# THE E-GAS TECHNOLOGY

## "E-GAS" GASIFICATION PROCESS HERITAGE



Developed Technology, Proto Plants & LGTI  
1973 - 1989



Spun off from Dow in 1989, 80% Dow Ownership  
Wabash River 1989-1997



NGC Corporation  
Purchased Destec from Dow  
in June 1997



DYNEGY

NGC changed its  
name  
to Dynegy in June 98



Global Energy acquired Dynegy's  
Gasification Assets in January 2000

2003

**ConocoPhillips**

Oct. 23, 2003

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## Multi-Fuel, Multi-Product Gasification Technology

### Feedstock Options

Coal  
Pet Coke  
MSW  
Sludge  
Biomass  
Waste oils  
Plastics

**Gasification  
Technology**

**Clean  
Low-cost  
Syngas**

Gasification Products  
• Synthetic Aggregate  
• Elemental Sulfur

### Product Options

GT/CC

Power  
Steam  
Hot  
Water

Chemical  
Production

Hydrogen  
CO  
Methanol  
Acetic Acid

F-T  
Synthesis

Diesel  
Naphtha  
Jet Fuel

## EGAS TECHNOLOGY HISTORY

PILOT PLANT	36 TPD	1975
PROTO 1	400 TPD	1979
PROTO 2	1600 TPD	1983
LGTI	2400 TPD	1987-1995
WABASH - coal	2550 TPD	1995- 2000
WABASH - petcoke	2000 TPD	2000-2003

Oct. 23, 2003

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# WABASH RIVER PLANT

Oct. 23, 2003

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## Wabash Facility Location



Oct. 23, 2003

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## Wabash River Project Overview

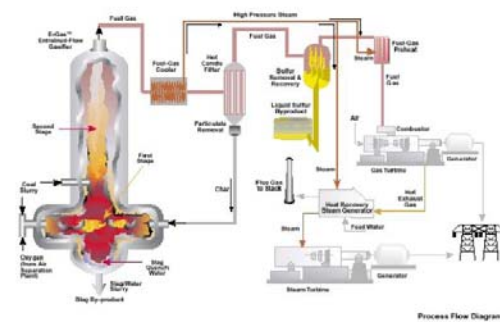
- E-Gas™ Technology Gasification Process
- Coal Gasification Combined Cycle Repowering
- Operational since 1995
- 262 MWe Net Output
- Bituminous Coal and Petcoke, up to 7 % S
- Cleanest Coal/Coke Fired Power Plant in the World

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## Wabash River Process Overview

- E- Gas™ Technology Gasification Process
- Oxygen Blown, Slurry Fed
- Continuous Slag Removal ( No lock hoppers)
- High Temperature Heat Recovery (~ 1000 DegC)
- Medium Temperature Dry Char Filtration (~ 450 DegC)
- COS Catalyst, MDEA Acid Gas Removal
- Claus based Sulfur Recovery
- Recycle of SRU Tailgas and Char to gasifier
- General Electric 7FA Gas Turbine, Dual Fuel (Oil/Syngas)



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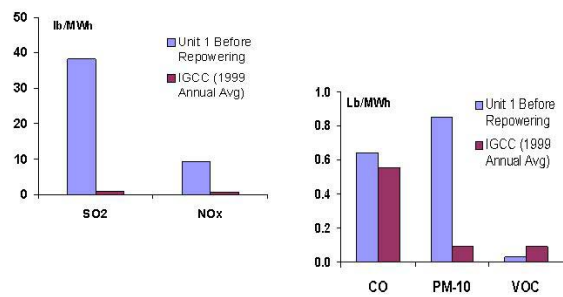
## Wabash River Energy Recognition

- Power Plant of the Year 1996 (Power Magazine)
- Power Plant Hall of Fame 2000 (Power Magazine)
- 1996 Certificate of Recognition for Energy Efficiency & Renewable Energy from the US DOE
- 1997 Certificate of Environmental Achievement from the National Awards Council for Environmental Sustainability
- 1998 Governor's Award for Excellence in Recycling
- Recognition in 2001 National Energy Policy
- Cleanest Coal/Coke Fired Power Plant in the World

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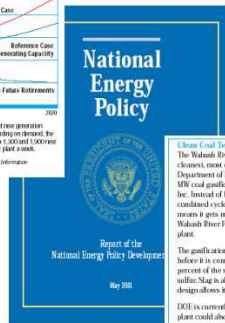
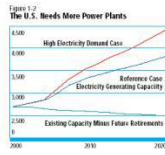
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## REPOWERING EMISSIONS COMPARISON



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**Clean Coal Technologies Up Close**

The Wabash River Coal Gasification Project in Terre Haute, Indiana, is one of the cleanest, most efficient coal burning facilities in the country. Partly funded by the Department of Energy (DOE) as part of its Clean Coal Technology Program, the 202 MW coal gasification facility is owned and operated by PSI Energy and Global Energy Inc. Instead of being directly burned, the coal is gasified and then combined in a combined cycle gas turbine. This allows the coal to burn more efficiently – which means it gets more energy than a traditional plant out of the same amount of coal. The Wabash River Facility is over 20 percent more efficient than a typical coal-fired power plant.

The gasification process also allows many of the impurities in the coal to be removed before it is combined to generate electricity. In the Wabash River project, over 90 percent of the sulfur is removed from the coal and marketed to industrial users of sulfur. Slag is also removed and is marketed to the construction industry. The plant's design allows it to burn other fuels, such as petroleum coke.

DOE is currently working with Global Energy and other industry partners to see if the plant could also be used to co-produce chemical feedstocks and transportation fuels. Additionally, DOE and its partners are studying lessons learned from the project to design a less expensive, more efficient coal gasification facility that would be ready for commercial deployment by 2005.

## E-Gas R&D

- Successful recent programs on petcoke, candle filters
- Currently involved in \$10 million of projects on methanol co-production, temperature measurement, process optimization studies, corrosion, hydrogen production, particulate removal
- Warm Gas Cleanup

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## TECHNOLOGY VISION

Year	1990-1999	2000-2009	2010-2020
Gasifier	Two Stage	Full Slurry Quench	Slurry Vaporization
Char Removal	Wet Scrubber	Dry Candles	Cyclone + Candle
Sulfur Removal		Amine	Warm Gas Cleanup
Mercury Removal			
ASU	Cryogenic		Membrane
Power Generation	Simple Cycle	7F IGCC	7FA
			7H
			Fuel Cell



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