# **SRI International**°

Final Report for NCCC • November 2016

# CO<sub>2</sub> CAPTURE FROM IGCC GAS STREAMS USING THE AC-ABC PROCESS

Final Report for National Carbon Capture Center, Wilsonville, AL (provided under a technology collaborative agreement) Covering the period July 1, 2014 through July 31, 2016 SRI Project P21321

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# CONTENTS

DISCLAIMER
ABBREVIATIONS
EXECUTIVE SUMMARY 6
INTRODUCTION
WORK PERFORMED
Small Pilot-Scale Design9
Integrated Plant Operation11
Analytical Testing15
System Modification and Maintenance
CO <sub>2</sub> / H <sub>2</sub> S Separation Process
Pilot-Plant Modification27
Pilot-Plant Operation
Discussion of Results
ILLUSTRATIONS
TECHNO-ECONOMIC ANALYSIS
CONCLUSION

## LIST OF FIGURES

Figure 1: Process flow diagram of the AC-ABC system.	10
<b>Figure 2</b> : CO <sub>2</sub> concentration in syngas (Run # 3).	11
<b>Figure 3</b> : CO <sub>2</sub> capture efficiency (Run # 3).	12
<b>Figure 4</b> : H <sub>2</sub> S concentration in syngas (Run # 3)	12
Figure 5: Absorber pressure (Run # 3)	14
Figure 6: Regenerator pressure (Run # 3).	15
<b>Figure 7</b> : CO <sub>2</sub> loading (Run # 2)	16
<b>Figure 8</b> : CO <sub>2</sub> loading (Run # 3)	17
<b>Figure 9</b> : CO <sub>2</sub> / NH <sub>3</sub> loading (Run # 3)	18
Figure 10: NH <sub>3</sub> & H <sub>2</sub> S concentrations in regenerator gas.	19
Figure 11: Gas composition (dry basis) of regenerated gas stream.	19
Figure 12: Mass balance of hydrogen.	20
<b>Figure 13</b> : NH <sub>3</sub> concentration in clean syngas	20
Figure 15: X-ray fluorescence analysis of the syngas-entrained particles	22
Figure 16: Semi-quantitative analysis by X-ray diffraction of syngas-entrained particles	23
Figure 17: Sulfur in outlet plenum of reactor intercooler HX-04.	24
Figure 18: Close-up view of sulfur in outlet plenum of reactor intercooler HX-04	25
Figure 19: Raw syngas availability	30
Figure 20: Syngas availability during Run # 10	31
Figure 21: Raw syngas, clean syngas, and the regenerated gas stream mass flow rate	32
Figure 22: Concentration of CO <sub>2</sub> in raw and clean syngas.	32
Figure 23: Concentration of H <sub>2</sub> S in raw and clean syngas.	33
Figure 24: Capture efficiency and CO <sub>2</sub> loading in solvent	34
Figure 25: CO <sub>2</sub> loading varied by changing solvent recirculation rate	35
Figure 26: Ammonia and sulfide concentrations in the lean solution	36
<b>Figure 27</b> : CO <sub>2</sub> loading in the lean and rich solutions	37
Figure 28: Ammonia in clean syngas from the absorber	37
Figure 29: Absorber bottom-stage temperature profile	38

Figure 30: Absorber and regenerator pressure.	39
Figure 31: Reboiler temperature for thermal siphon	. 39
Figure 32: Regenerated gas stream main constituents.	40
Figure 33: Methane and carbon monoxide in the regenerated stream.	41
Figure 34: Hydrogen and argon in the regenerated gas stream	41
Figure 35: Gas mass balance	42
Figure 36: Energy input to reboiler	43
Figure 37: BPSC regeneration temperature chart.	45
<b>Figure 38</b> : The outlet plenum of the BPSC regeneration cooler (sulfur condenser) – close-up view after the second test campaign.	. 46
<b>Figure 39</b> : The outlet plenum of the BPSC regeneration cooler after the second test campaign (sulfur condenser).	. 46
Figure 40: A photograph of the AC-ABC pilot plant at the NCCC	47
Figure 41: A photograph of the process skids	47
Figure 42: A photograph of the sulfur reactors	48
Figure 43: A photograph of skid B-1 with the sulfur condensers	48
Figure 44: A photograph of the electrical and control panels	. 49

### LIST OF TABLES

<b>Table 1</b> : Typical raw syngas stream composition from the lignite coal gasifier at NCCC	29
<b>Table 2</b> : CO <sub>2</sub> capture costs compared to the IGCC base case (Case B5A)	50
<b>Table 3</b> : Capital cost comparison to the IGCC base case (Case B5A).	51

# **ABBREVIATIONS**

AC-ABC	Ammonium carbonate - ammonium bicarbonate
AGR	Acid gas removal
BPSC	Bechtel pressure-swing Claus
BTU	British thermal unit
$CH_4$	Methane
CO	Carbon monoxide
$CO_2$	Carbon dioxide
COE	Cost of electricity
DI	Deionized
DOE	Department of Energy
EPDM	Ethylene propylene diene monomer
FTIR	Fourier transform infrared
GC	Gas chromatograph
GWh	Gigawatt hour
H <sub>2</sub>	Hydrogen
$H_2S$	Hydrogen sulfide
ID	Internal diameter
IGCC	Integrated gasification combined cycle
IR	Infrared
Kw	Kilowatt
KWh	Kilowatt hour
NCCC	National Carbon Capture Center
NETL	National Energy Technology Laboratory
PC	Pulverized coal
PID	Proportional-integral-derivative
SCU	Syngas cleanup unit
SRI	SRI International
TOC	Total overnight cost
UV/Vis	Ultraviolet visible
WGS	Water-gas shift
WGSR	Water-gas-shift reactor
XRD	X-ray diffraction

#### **EXECUTIVE SUMMARY**

SRI International (SRI), in collaboration with EIG, Inc. and Bechtel Hydrocarbon Solutions, Inc., is performing a multi-year effort to develop a promising technology to capture carbon dioxide (CO<sub>2</sub>) from coal-fired power plants. The overall objective of the proposed program is to develop, for integrated gasification combined cycle (IGCC)-based power plants, an innovative, low-cost CO<sub>2</sub> capture technology based on absorption on a high-capacity and lowcost aqueous ammoniated solution. The specific objectives are to: (1) test the technology on a bench-scale reactor to validate the concept and to determine the optimum operating conditions for a pilot-scale reactor; (2) design, build, and perform tests using the pilot-scale reactor capable of continuous integrated operation; and (3) perform a technical and economic evaluation of the technology.

Bench-scale experimental results carried out in Budget Period 1 led to the design of an integrated small-scale pilot plant with a single absorber column for simultaneous  $CO_2$  and hydrogen sulfide (H<sub>2</sub>S) absorption from syngas and a single regenerator column to strip  $CO_2$  and H<sub>2</sub>S. The Bechtel pressure-swing Claus (BPSC) process was designed by Bechtel downstream of SRI's ammonium carbonate – ammonium bicarbonate (AC-ABC) process to treat H<sub>2</sub>S/CO<sub>2</sub> regenerated gas stream and convert H<sub>2</sub>S to elemental sulfur. The combined system was installed at National Carbon Capture Center (NCCC), Wilsonville, Alabama in 2014-2015 and operated in September/October 2015 during the G2 run of the air blown gasifier and again in April / May 2016 during the G4 run.

During the two test campaigns of the small pilot-scale setup of the integrated AC-ABC process, over 700 hr of test data was collected during 10 runs including a continuous run of 175 hrs.

Based on the results from the test runs and liquid and gas analysis, the following conclusions can be drawn:

- Ammoniated solution is very effective in rapid absorption of CO<sub>2</sub> with high CO<sub>2</sub> loading at elevated pressure. CO<sub>2</sub> capture efficiency greater than 99% was demonstrated, with 10 – 12% CO<sub>2</sub> effective loading.
- H<sub>2</sub>S can be simultaneously absorbed along with CO<sub>2</sub> in a single absorber column. The H<sub>2</sub>S capture efficiency was greater than 99%.
- The absorption and thus loss of fuel gas species like hydrogen (H<sub>2</sub>), carbon monoxide (CO), and methane (CH<sub>4</sub>) was shown to be very low.
- Low ammonia losses from the system were demonstrated.

- Both the absorber and regenerator were operated at similar elevated pressure, thus reducing the need for pumping solvents across pressure boundaries.
- Simultaneous stripping of CO<sub>2</sub> and H<sub>2</sub>S was demonstrated in a single column at elevated pressure and moderate temperature.
- The BPSC process demonstrated conversion of H<sub>2</sub>S to high-purity elemental sulfur, and a clean CO<sub>2</sub> gas stream at elevated pressure was available for sequestration or transportation.
- Availability of regenerated CO<sub>2</sub> at elevated pressure significantly reduces the compressor requirements.
- The cost of CO<sub>2</sub> capture using AC-ABC / BPSC process from IGCC gas stream is less than \$30/metric ton of CO<sub>2</sub>

#### **INTRODUCTION**

Capturing CO<sub>2</sub> from coal-fired power plants is a critical step in carbon sequestration. In the IGCC process, which generates electricity more efficiently than pulverized coal (PC) combustion power plants, coal is reacted with steam and oxygen under pressure in the range of 300 to 1000 psi to form a fuel gas containing mainly CO, H<sub>2</sub>, H<sub>2</sub>S, CO<sub>2</sub>, and residual steam. Carbon monoxide in the gas stream is converted to CO<sub>2</sub> and H<sub>2</sub> by using the water-gas shift reaction at about 200° to 285°C. The gas stream leaving the water-gas-shift reactor (WGSR) contains mainly H<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>S, and H<sub>2</sub>O. A H<sub>2</sub>-rich fuel gas suitable for combustion in a gas turbine is produced by condensing the steam and removing the CO<sub>2</sub> and H<sub>2</sub>S. The current "bestcase" option for carbon capture is using a liquid solvent such as Selexol or Rectisol to absorb CO<sub>2</sub> and H<sub>2</sub>S at elevated pressures.

The AC-ABC process for capture of  $CO_2$  and  $H_2S$  in the pre-combustion gas stream offers many advantages over other solvent-based technology. The process relies on the simple chemistry of the  $NH_3$ - $CO_2$ - $H_2O$ - $H_2S$  system, and on the ability of the aqueous ammoniated solution to absorb  $CO_2$  and  $H_2S$  at near ambient temperatures and to release it as a high-pressure gas at a moderately elevated temperature.

SRI, in collaboration with EIG, Inc. and Bechtel Hydrocarbon Technology Solutions, Inc., has performed a multi-year effort to develop this promising technology. The overall objective of the proposed program was to develop—for IGCC-based power plants—an innovative, low-cost CO<sub>2</sub> capture technology based on absorption on a high-capacity and lowcost aqueous ammoniated solution. The specific objectives were to:

- (1) Test the technology on a bench-scale batch reactor to validate the concept and to determine the optimum operating conditions for a small pilot-scale reactor,
- (2) Design, build, and perform tests using a small pilot-scale reactor capable of continuous integrated operation, and
- (3) Perform a technical and economic evaluation of the technology.

The experimental program consisted of three tasks:

- 1. Bench-scale batch tests at SRI
- 2. Pilot-scale integrated, continuous tests at the NCCC
- 3. Project management

#### WORK PERFORMED

#### **Small Pilot-Scale Design**

Based on the data obtained from bench-scale batch testing, a small pilot plant was designed to handle 500-lb/hr syngas from the syngas cleanup unit (SCU) at the NCCC. Construction of the small-scale pilot plant was completed at the NCCC during spring / summer 2015. An integrated system was designed (see Figure 1) and installed with a single absorber column for simultaneous capture of  $CO_2$  and  $H_2S$  from syngas and a single regenerator for simultaneous stripping of  $CO_2$  and  $H_2S$  at moderate temperature and elevated pressure. A compressor was used to increase the syngas pressure supplied from air blown gasifier at the NCCC. The syngas compressor was equipped with a variable frequency drive to enable syngas mass flow control to the absorber.

The absorber was comprised of an 8" internal diameter (ID), 40-foot tall stainless steel column with three absorption stages. Commercially available stainless steel structural packing was used in the first and second absorption stages for gas-liquid contacting. The third stage used bubble cap trays, also fabricated out of stainless steel. The purpose of third absorption stage was to control ammonia emission from the system. A Horiba  $CO_2$  analyzer based on infrared (IR) technology was used to monitor  $CO_2$  concentration in the incoming syngas.

Two positive displacement pumps were used to circulate the solvent in each of the first and second stages of absorption. A third positive displacement pump was used to deliver fresh lean solution to the top of the third absorption stage. The bottom of absorber column was maintained at 120°F using heat tracing to prevent precipitation.

An 8" ID and 40-foot tall water-wash column was installed downstream of the absorber to capture ammonia from the clean syngas using deionized (DI) water. The water-wash column was equipped with one stage of structural packing and a second stage of bubble cap trays to minimize ammonia emissions. An automated back-pressure control valve was used to maintain desired pressure in the absorber and water-wash columns. A positive displacement pump delivered DI water to the top of water-wash column, while another positive displacement pump was used to circulate the water through the first absorption stage. A Horiba  $CO_2$  analyzer was used to monitor  $CO_2$  concentration in the clean syngas. A Fourier transform infrared (FTIR) spectrometer was installed to monitor ammonia loss in clean syngas.

A slip stream of rich solution was taken out of the first absorption stage at the bottom of the absorber column recirculation loop using a level controller and an automatic control valve. The level controller maintained rich solution inventory in the bottom of the absorber column, and

9

the slip stream was delivered to the regenerator for stripping  $CO_2$  and  $H_2S$ . The rich solution delivery lines were heat traced to prevent precipitation.

The regenerator column was equipped with a reboiler to use moderate pressure steam and strip  $CO_2$  and  $H_2S$  from the rich solution. The regenerator column was designed with 8" ID and 40-foot tall stainless steel column. The top stage of the regenerator contained the water wash for stripped gas stream to reduce ammonia emission. An automated back-pressure control valve maintained the stripping pressure in the regenerator column.

The solvent is transferred from the absorber to regenerator using a small pressure differential between the two columns.

The water-wash waste stream from water-wash and regenerator columns was collected into a spent water tank for disposal. In a larger system, the waste water will be sent to a sourwater stripper to recover ammonia. Chemical analysis of the samples from liquid waste streams and the lean solution demonstrated low loss of ammonia during normal operation of the pilot plant.



Figure 1: Process flow diagram of the AC-ABC system.

#### **Integrated Plant Operation**

The small-scale AC-ABC pilot plant was operated using slipstream of up to 500 lb/hr syngas from air blown gasifier at the NCCC. Lignite coal with high sulfur content was used in the gasifier resulting in ~ 2000 ppmv hydrogen sulfide in the syngas slipstream. A shift reactor operated by the NCCC converted carbon monoxide to carbon dioxide using high-pressure steam. Syngas obtained after the shift reactor and quench typically had 14 - 15% CO<sub>2</sub>, 0.2% H<sub>2</sub>S, 1% CO, 12% H<sub>2</sub>, 0.5% O<sub>2</sub>, and 0.5% CH<sub>4</sub> with the balance as nitrogen. Syngas was supplied at 60°F -70°F and 180 psig. During upset conditions of syngas clean-up unit, the slipstream of syngas was replaced with nitrogen by the NCCC. A total of five (5) test runs (Run # 1 to Run # 5) were carried out during first test campaign in 2015, collecting over 300 cumulative hours of test data.

A syngas compressor, which was part of the AC-ABC pilot plant, was equipped with a variable frequency drive used to control the syngas mass flow through the AC-ABC absorber column. Syngas flow was varied between 300 - 450 lb/hr at 450 psig. An IR-based CO<sub>2</sub> analyzer was used to measure CO<sub>2</sub> in the syngas slip stream on a dry basis. In addition, the NCCC monitored composition of syngas after the shift reactor and provided the syngas composition data. A high-pressure, positive displacement pump was used to feed the lean solution at the top of the absorber column. The pump was equipped with a variable frequency drive to control the solvent flow through the system. The lean solvent flow was varied between 500 - 1000 lb/hr.



**Figure 2**: CO<sub>2</sub> concentration in syngas (Run # 3).



**Figure 3**: CO<sub>2</sub> capture efficiency (Run # 3).



Figure 4: H<sub>2</sub>S concentration in syngas (Run # 3).

The bottom section of the absorber column was used as a reservoir for the rich solution. A positive displacement pump circulated the rich solution through the first absorption stage of the absorber. The reservoir and first stage of the absorber were heat traced, and the temperature was maintained at  $120^{\circ}$ F to prevent precipitation of solids from the CO<sub>2</sub>-rich solution. A positive displacement pump was used to circulate the solvent through a water-cooled heat exchanger with a bypass control to maintain the temperature and maximize CO<sub>2</sub> capture in this section. Both the first- and second-stage absorbers are comprised of stainless steel structural packing.

The third stage of the absorber was equipped with bubble cap trays. The purpose of the trays was to minimize ammonia loss by controlling the lean solution and semi-rich solution flow rates.

Clean syngas was obtained after the capture of  $CO_2$  and  $H_2S$  and was passed through a water-wash column to remove ammonia. An automated back-pressure control valve at the exit of water-wash column was used to control the absorption pressure in the absorber.

Demineralized water was added to the top of the water-wash column using a positive displacement pump to reduce the ammonia emission. An IR-based  $CO_2$  analyzer was used to monitor  $CO_2$  in the clean syngas to determine efficacy of  $CO_2$  absorption process. In addition, an FTIR spectrometer was installed to monitor ammonia content in the clean syngas stream. The clean syngas composition was monitored by the NCCC using a gas chromatograph (GC) on a continuous basis.

As the lean solution was added to the top of the absorber, an automated level control valve sent a side stream of the rich solution to the regenerator column. All rich-solution lines, instrumentation, and control valves were heat traced to prevent precipitation of solids.

Liquid samples were collected from the water-wash waste stream to monitor ammonia loss.

During an upset condition, particles were entrained with raw syngas. These particles were analyzed and found to be 1 - 100 micron in size and mainly contained calcium, magnesium, iron, and chromium as constituents. The syngas compressor was equipped with an inlet filter that was sized to remove 100-micron or larger particles from the gas stream. The particles clogged several control valves, and the pilot plant had to be shut down. After removing the solvent, the system was rinsed several times to clean the control valves, lines, and columns, and the system was restarted.



Figure 5: Absorber pressure (Run # 3).

The rich solution side stream to the regenerator was passed through a heat recovery heat exchanger to recover heat from the lean solution. Steam at 175 psig was supplied to the reboiler attached to the regenerator. Steam flow was controlled using a manual control valve. As expected, a thermal siphon developed between regenerator and reboiler. Part of the rich-solution stream was split from the main stream and fed to the middle stage of regenerator to reduce ammonia loss from the regeneration process. A water-wash stage was added to the top of the regenerator column to remove ammonia from regenerated gas stream using demineralized water. Liquid samples were collected from the water-wash waste stream and monitored for ammonia content.



Figure 6: Regenerator pressure (Run # 3).

An automated back-pressure control valve was used to maintain the regenerator pressure, and the regenerated stream was sent to the BPSC skid for converting  $H_2S$  to elemental sulfur. The level controller maintained the liquid level in the regenerator bottom section, and the lean solution was returned to the lean solution surge tank through a heat recovery heat exchanger.

The reboiler was operated at  $290^{\circ}F - 310^{\circ}F$  by controlling the steam input and maintaining a back pressure of 300 - 350 psig.

#### **Analytical Testing**

Liquid samples were collected periodically from the lean solution stream and analyzed for ammonia and CO<sub>2</sub> loading. A target CO<sub>2</sub>/NH<sub>3</sub> loading ratio for the lean solution was set at 0.4 - 0.45. The CO<sub>2</sub> loading of ~ 100 g/liter of water was demonstrated under one operating condition [Figure 7: CO<sub>2</sub> loading (Run # 2)] during the first test campaign.



Figure 7: CO<sub>2</sub> loading (Run # 2).

As mentioned earlier, syngas was replaced by nitrogen during the upset conditions of the gasifier and/or SCU at the NCCC. This caused a system upset in AC-ABC pilot plant and a loss of ammonia from the solvent [Figure 8.  $CO_2$  loading (Run # 3)]. A modified operating procedure was implemented to minimize ammonia loss in the second test campaign.



Figure 8: CO<sub>2</sub> loading (Run # 3).

Based on the lean solution analysis, the  $CO_2/NH_3$  ratio was determined to be at 0.4 – 0.45. Rich-solution loading was calculated as 0.7 [Figure 9:  $CO_2 / NH_3$  loading (Run # 3)]. With heat tracing in place, no appreciable solids precipitation was noticed in the rich-solution path. A higher  $CO_2$  loading in the rich solution of 0.8 and a lower loading of 0.35 in the lean solution was targeted for the second test campaign to improve  $CO_2$  loading in solvent up to 150g/liter or higher.



**Figure 9**: CO<sub>2</sub> / NH<sub>3</sub> loading (Run # 3).

The regenerated gas stream was analyzed for  $H_2S$  content using Draeger tubes.  $H_2S$  varied between 0.7 to 1.5% in the regenerated stream (Figure 10:  $NH_3 \& H_2S$  concentration in regenerator gas).



Figure 10: NH<sub>3</sub> & H<sub>2</sub>S concentrations in regenerator gas.

A pressure-sampling vessel was used to collect gas samples from the regenerated gas stream and analyzed at the NCCC using a GC. The gas analysis demonstrated a very low level of fuel gas species in the regenerated gas stream as shown in Figure 11 [Gas composition (dry basis) of regenerated gas stream], thus there was a negligible loss of fuel gas species from syngas, (Figure 12 Mass balance of hydrogen).

10/13/2	2015 14:	30	10/15/2	2015 9:30
H2	0.07	%	H2	0.08%
N2	1.56	%	N2	0.45%
СО	0.02	%	CO	0%
CH4	0	%	CH4	0%
Ar/02	0.26	%	Ar/02	0.01 %
H2S	1.42	%	H2S	1.56%
CO2	Bal.	%	CO2	Bal. %
COS	25	ppm	COS	19 ppm

Figure 11: Gas composition (dry basis) of regenerated gas stream.



Figure 12: Mass balance of hydrogen.



Figure 13: NH<sub>3</sub> concentration in clean syngas.

The ammonia concentration was 600 ppm in the clean syngas stream (Figure 13) as measured using Draeger tubes. This level of ammonia was caused by failure of the bubble cap trays in the third stage of the absorption column.

#### **System Modification and Maintenance**

A high-pressure positive displacement pump for the lean solution failed during operation. The diaphragm was replaced, and the pilot plant was restarted. The probable cause of diaphragm failure was either a pressure surge or the presence of syngas-entrained particles.

As described earlier, particles were entrained with syngas early in the test campaign, and accumulated in the solvent. The particles were recovered from the liquid stream and tested for size and composition (Figures 14, 15, and 16). The origin of the particles has not been determined.



Figure 14: Entrained particles – size distribution.

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Customer Account: DTNCCCC

Customer ID Number: PSDF

Sample Date: 9/29/2015

Delivery Date: 9/30/2015

# CERTIFICATE OF ANALYSIS

To: Ms. Wynema Kimbrough PSDF P.O. Box 1069 Wilsonville, AL 35186

Description: PSDF # AB36988 Powder from Absorber Bottom

Laboratory ID Number: AV22912

Name	Reference	Results	Units
Aluminum Ovide	Comment	0.18	percent
Arsenic Oxide	XRF (E. Scan)	Not Detected	percent
Calcium Oxide	XRE (E. Scan)	57 60	percent
Cobalt Oxide	XRE (E. Scan)	0.02	nercent
Chromium Oxide	XRE (E. Scan)	3.11	percent
Copper Oxide	XRF (E. Scan)	0.07	percent
Iron Oxide	XRF (E. Scan)	4.84	percent
Magnesium Oxide	XRF (E. Scan)	27.66	percent
Manganese Oxide	XRF (E. Scan)	0.37	percent
Molybdenum Oxide	XRF (E. Scan)	0.06	percent
Sodium Oxide	XRF (E. Scan)	0.03	, percent
Niobium Oxide	XRF (E. Scan)	0.01	percent
Nickel Oxide	XRF (E. Scan)	0.43	percent
Phosphorus Pentoxide	XRF (E. Scan)	0.18	percent
Silicon Oxide	XRF (E. Scan)	1.09	percent
Strontium Oxide	XRF (E. Scan)	0.04	percent
Sulfur Oxide	XRF (E. Scan)	4.19	percent
Titanium Oxide	XRF (E. Scan)	0.07	percent
Zinc Oxide	XRF (E. Scan)	0.02	percent
Zirconium Oxide	XRF (E. Scan)	0.02	percent

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Page 1 of 1

Figure 15: X-ray fluorescence analysis of the syngas-entrained particles.

Customer Account: Sample Date: Customer ID Number: Delivery Date:	DTNCCCC 9/29/2015 PSDF 9/30/2015
Sample Date: Customer ID Number: Delivery Date:	9/29/2015 PSDF 9/30/2015
Customer ID Number: Delivery Date:	PSDF 9/30/2015
Delivery Date:	9/30/2015
Results	Units
-Ray Diffraction.	
	-Ray Diffraction.

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Figure 16: Semi-quantitative analysis by X-ray diffraction of syngas-entrained particles.

#### CO<sub>2</sub> / H<sub>2</sub>S Separation Process

A BPSC process, designed by Bechtel was installed downstream of the AC-ABC system to separate  $H_2S$  from CO<sub>2</sub> regenerated gas stream and convert the same to elemental sulfur using SO<sub>2</sub>. There were several design, installation, and operational shortcomings in the BPSC system. A positive displacement pump was used to provide liquid SO<sub>2</sub> at a calculated rate based on  $H_2S$ concentration in the regenerated stream. Due to the small flows involved, it was difficult to control the pump output to match the desired demand. As shown in Figures 17 and 18, a significant amount of sulfur was recovered in elemental form from the regenerated gas stream, even after all the system problems. However, inadequate heat tracing prevented the flow and quantification of sulfur produced.



Figure 17: Sulfur in outlet plenum of reactor intercooler HX-04.



Figure 18: Close-up view of sulfur in outlet plenum of reactor intercooler HX-04.

#### Second Test Campaign, 2016

Based on the data from the first test campaign and operating experience, the following objectives were targeted for the second test campaign:

1. **Objective**: Achieve steady, continuous operation for 100 hours or more at 99+% CO<sub>2</sub> and H<sub>2</sub>S capture efficiency, regeneration, and sulfur production.

**Results**: A total of over 400 hr of operating data was collected from the second test campaign, including a 175-hr (7 days) continuous integrated run. The test results show that both  $CO_2$  and  $H_2S$  can be captured in a single absorber operating at 350 psig, 120°F. A high capture efficiency for both  $CO_2$  and  $H_2S$  (> 99+ %) was demonstrated during this test campaign. This test campaign had > 80% uptime for the pilot plant.

 $CO_2$  in clean syngas was measured at 500 – 1000 ppm with absorption at 350 psig. Operation at higher pressure will further reduce the  $CO_2$  emission. H<sub>2</sub>S was reduced from 2000 ppm in the inlet gas stream to < 2 ppm (below the detection limit of the GC) in the clean syngas stream.

A stream of > 98+ mol% CO<sub>2</sub> and 1.5 mol% H<sub>2</sub>S was regenerated at 350 psig from a single regenerator. The downstream process demonstrated high conversion of H<sub>2</sub>S to high-purity elemental sulfur. However, due to heat tracing problems, sulfur production could not be quantified.

2. **Objective**: Achieve >  $100 \text{ g CO}_2$  loading per  $1000 \text{ g water using an 8-molal NH}_3$  solution.

**Results**: An effective loading of 120 g  $CO_2/1000$  g water was demonstrated under steady-state operation during the test campaign with maximum loading, and 140 g  $CO_2/1000$  g water was achieved.

3. **Objective**: Optimize operation of top of absorber to minimize NH<sub>3</sub> flow to water wash.

**Results**: Bubble cap trays in the top absorber section were replaced by structural packing for this test campaign, and later the top absorber section was bypassed due to ineffective liquid-to-gas contact. The absorber was operated with only two stages, and still the test results show high  $CO_2$  capture efficiency. Ammonia emission from the absorber was measured at 500 – 1000 ppm under steady operating conditions. During operation at higher pressure and with the third-stage absorption, ammonia emissions can easily be reduced to a very low value (~ 20 ppm). In a commercial system, ammonia will be recovered in a sour-water stripper.

4. **Objective**: Optimize water-wash operation to demonstrate low water use.

**Results**: Based on lab tests carried out at atmospheric pressure at SRI International, the bubble cap trays were modified to the eliminate flooding experienced during the first test campaign. However, the modified bubble cap trays did not perform as expected, and low water use could not be demonstrated. ASPEN modeling shows it is possible to capture ammonia emissions from the absorber with minimal water use when properly designed bubble cap trays are used. Commercially designed bubble cap trays will be included in a future test campaign to demonstrate low water use in the water-wash column.

5. **Objective**: Operate regenerator steam in steady mode and operate the lean solution with  $CO_2$  loading < 0.35.

**Results**: The regenerator was operated with steady steam flow and a low  $CO_2$  loading in the lean solution (< 0.35  $CO_2/NH_3$  ratio) was demonstrated. It is possible to automate steam flow control using a reboiler temperature feedback loop control. The same will be considered for a future test campaign.

6. **Objective**: Operate BPSC skid with controlled  $SO_2$  flow and quantify sulfur formation.

**Results**: The sulfur dioxide delivery system was modified to enable controlled flow of  $SO_2$  to the process system. The test results show high conversion of  $H_2S$  to high-purity elemental sulfur. However, due to problems with heat tracing, liquid sulfur did not flow to the collection vessel and instead froze at cold spots in lines and/or valves and hence could not be quantified. A future test campaign could use higher temperature heat tracing and better insulation.

#### **Pilot-Plant Modification**

Based on operational experience, data collected, and lessons learned from the first test campaign during September/October 2015, the following modifications were carried out prior to the second test campaign at the pilot plant.

- 1. During the first test campaign, some solid particles were entrained with raw syngas. An inlet syngas particle filter with 10-micron mesh size was installed upstream of the syngas compressor to remove any solids entrained in the raw syngas and prevent damage to the compressor.
- 2. Heat tracing and insulation on the system process piping and vessels was improved to prevent precipitation from the rich solution during the process upset conditions. The gassampling lines were heat traced to prevent precipitation from the gas phase.
- 3. It had been a challenge to source commercially designed bubble cap trays for the small diameter of the pilot process vessels for the AC-ABC process. To demonstrate low water use

for ammonia emission control, the bubble cap trays are the most efficient gas-liquid contactors. During the first test campaign, in-house designed and fabricated bubble cap trays were installed. During the pilot plant operation, tray flooding was observed and the trays were bypassed during extended operation. Bubble cap trays were field-modified based on atmospheric pressure tests carried out at the SRI lab. The modified bubble cap trays did not perform as intended in the water-wash column and had to be bypassed again. For a future test campaign, we expect that commercial bubble cap trays will be used, and a potential source for them has been identified.

- 4. The absorber top stage also contained bubble cap trays to effectively control ammonia emissions from the absorber by adjusting the ratio of the lean solution feed rate to a recirculation flow rate. The first test campaign resulted in bubble cap trays flooding and solvent carryover to water-wash column. The top absorber stage bubble cap trays were replaced by structural packing. Due to low liquid loads, the structural packing was not effective. The third stage of absorber was bypassed, and the absorber was operated with two working stages. Test results show that capture efficiency was still very high for both CO<sub>2</sub> and H<sub>2</sub>S, even with only two operating stages. The recorded ammonia emissions were between 500 1000 ppmv.
- 5. Steam-cleaning valves were added to the piping in which clogging was experienced due to process upset conditions during the first test campaign. This enabled faster turnaround between two test runs and resulted in > 80% uptime for the pilot plant during the second test campaign.
- 6. The solvent circulation pump's Viton diaphragms were replaced with ethylene propylene diene monomer (EPDM) material, which is more chemically compatible with sulfides in the solvent.
- 7. Thermal mass flow meters in the exit gas lines were replaced by a Coriolis meter for better data collection in the gas flows.
- The particles entrained in raw syngas during the first test campaign damaged the FTIR mirrors. Hence, ammonia emissions could not be monitored during the first test campaign. The FTIR spectrometer was repaired and installed to monitor ammonia in the gas outlet from the absorber column.
- 9. A gas heater was added to provide heated nitrogen for regeneration of sulfur from the catalytic bed.

- 10. The SO<sub>2</sub> delivery system was modified, and a pulsation dampener was added to the SO<sub>2</sub> line to reduce flow fluctuations due to diaphragm pump. A new SO<sub>2</sub> vaporizer was installed to convert liquid sulfur dioxide to vapor. Under the modified arrangement for SO<sub>2</sub> delivery, a recirculation loop was added to the pump and a Coriolis-based flow control was designed to withdraw the desired amount of liquid from the loop. The performance of the SO<sub>2</sub> delivery system improved somewhat, but further improvements can be made by replacing the pump with one that has a higher flow rate such that the recirculation loop always has more than the desired quantity of liquid.
- 11. Steam tracing was added to the sulfur product cooler/melter to provide faster heating and cooling steps. Electric tracing was deemed not fast enough for the heat-up step. By using steam in the coil around the vessels, the desired temperature was achieved in a short time. During the cooling step, steam was replaced with cooling water to reduce the shell temperature of the vessel in a short period of time.

The above-mentioned modifications significantly improved the pilot operation during the second test campaign. During the April/May 2016 test campaign, five test runs were carried out with a total of 400 hr of data collected from 480 hr of syngas availability from the gasifier. This campaign included one continuous run of 175 hr (7 days).

#### **Pilot-Plant Operation**

The second test campaign of the AC-ABC / BPSC pilot plant was carried out at the NCCC in Alabama in April/May 2016 and included Run # 6 to Run # 10. The NCCC operated the coal gasifier using PRB, a mix of PRB/lignite, and lignite coal. The SCU at the NCCC provided 500 lb/hr shifted syngas after tar removal at 170 psig and  $50 - 60^{\circ}$ F. Shifted syngas contained  $12 - 14 \mod 6 \operatorname{CO}_2$  and  $1800 - 2000 \operatorname{ppmv} \operatorname{H}_2S$ . Availability of raw syngas from the NCCC gasifier was hampered by gasifier maintenance issues, Figure 19. The gasifier had to be shut down for maintenance a few times during the test campaign for multiple days at a time. The raw syngas with  $\operatorname{H}_2S > 1000 \operatorname{ppmv}$  was available for ~ 480 hours during the G4 test campaign.

Syngas Temp	Syngas Pressure	Ar	$N_2$	СО	CO <sub>2</sub>	$CH_4$	$H_2$	$H_2S$
TI9860	PI9863	AI2603A	AI2603B	AI2603C	AI2603D	AI2603E	AI2603G	AI2603J
۴F	psig	%	%	%	%	%	%	ppm
67.4	170.2	0.6	74.4	0.03	13.4	0.6	10.1	2081.7

**Table 1:** Typical raw syngas stream composition from the lignite coal gasifier at NCCC.



Figure 19: Raw syngas availability.

A syngas compressor was used to increase the pressure of raw syngas to 350 psig. The compressor is equipped with a variable speed drive to control the flow of syngas to the absorber. A particle filter was added to the compressor inlet, and a compressor bypass line was installed to vent any residual gases in the syngas supply line. IR-based CO<sub>2</sub> monitors were used to measure CO<sub>2</sub> concentration in the inlet raw syngas and clean syngas. In addition, the NCCC provided raw syngas composition and also measured clean syngas composition using a GC. A separate GC measured H<sub>2</sub>S in the clean syngas. The repaired FTIR spectrometer was used to monitor ammonia concentration in the gas stream exiting the absorber. Coriolis meters were used measure mass flow of raw and clean syngas and regenerated stream. Grab samples of regenerated gas stream were taken periodically and analyzed for composition by the NCCC using a GC.

#### **Discussion of Results**

Detailed data and analysis are presented here for Run # 10, during which the pilot plant was operated for ~ 175 hr (7 days) on a 24-hr basis, Figure 20. During the run, some ammoniated solution was removed and fresh solution was added to the system. There was some interruption to syngas during this run; the pilot plant was put in a hot-standby mode during which solvent circulation continued but the steam was cutback. The inlet and outlet syngas streams were

closed. Another interruption was caused by back pressure in the syngas return line, probably due to clogging. The inlet syngas was temporarily stopped to clear the clog, and the pilot operation was continued.



Figure 20: Syngas availability during Run # 10.

During Run # 10, raw syngas flow was maintained at 300 lb/hr with a back pressure regulated in the water-wash column at 350 psig. Near the very end of the run, raw syngas flow rate was increased to 350 lb/hr to demonstrate high  $CO_2$  loading. Figure 21 below shows mass flow rates of the three gas streams: raw syngas, clean syngas, and the regenerated gas stream. All gas flows were monitored using Coriolis flow meters.



Figure 21: Raw syngas, clean syngas, and the regenerated gas stream mass flow rate.

Raw syngas contained 14 - 15 mol% CO<sub>2</sub> and ~ 2000 ppm H<sub>2</sub>S. Figure 22 shows the concentration of CO<sub>2</sub> in raw and clean syngas, and Figure 23 shows the H<sub>2</sub>S concentration in raw and clean syngas.



Figure 22: Concentration of CO<sub>2</sub> in raw and clean syngas.



Figure 23: Concentration of H<sub>2</sub>S in raw and clean syngas.

Raw syngas was introduced in the absorber first stage. Semi-rich solvent was circulated at the bottom of the absorber with no external cooling. A proportional-integral-derivative (PID) controller and back-pressure regulating valve was used to maintain pressure in the absorber and water-wash column. After being scrubbed in the water-wash column, clean syngas was returned to the thermal oxidizer with a gas sampling line to a GC to monitor the gas composition. Solvent circulation was maintained using a level sensor in the absorber reservoir and a level control valve. Rich solution from the bottom of the absorber was sent to regenerator.

The regenerated gas stream was either returned to thermal oxidizer or sent to the sulfur conversion unit. Grab samples from the regenerated gas stream were tested by the NCCC to determine gas composition.

Bubble cap trays in the top stage of absorber flooded during the first test campaign due to inadequate design. The trays were replaced by structural packing for the second test run. The packing did not perform well due to low liquid loads and was bypassed during the test runs. The absorber was operated with two stages and achieved > 99.5 % CO<sub>2</sub> capture and > 99.5 % H<sub>2</sub>S capture in a single vessel as shown in Figure 24.

During a period from 30 to 84 hr, the capture efficiencies for both  $CO_2$  and  $H_2S$  were marginally lower due to low ammonia concentration in the solvent. Fresh ammonia solution was added to the system resulting in high capture efficiency for both  $CO_2$  and  $H_2S$ . Effective  $CO_2$  loading was calculated based on the mass of  $CO_2$  captured and the water content of the circulating solution. The solvent circulation rate was varied to change the effective loading of  $CO_2$  in solvent.



Figure 24: Capture efficiency and CO<sub>2</sub> loading in solvent.



Figure 25: CO<sub>2</sub> loading varied by changing solvent recirculation rate.

Ammonia concentration in the circulating solvent and the CO<sub>2</sub> and sulfide loadings were determined by wet analysis of liquid samples in the lab. Figure 26 shows ammonia concentration and sulfide concentration in the lean solution, and Figure 27 shows the CO<sub>2</sub>-to-ammonia concentration in the lean solution. A titrator was used to determine ammonia and CO<sub>2</sub> loadings in the lean solution, and the same is calculated for the rich solution. An ultraviolet visible (UV/Vis) spectrometer was used for sulfide concentration determination. Sulfides were stabilized in the liquid samples using a buffer, and color was developed using methylene blue. Sulfide was observed at a 665-nm wavelength.



Figure 26: Ammonia and sulfide concentrations in the lean solution.

Relative  $CO_2$  loading in the lean and rich solutions determines the effectiveness of the  $CO_2$  capture process. The titration method of liquid sampling to determine relative concentration can be replaced by an online concentration measurement system. Online measurement can guide the operation to improve effective  $CO_2$  loading without precipitation and optimize steam use in the reboiler where  $CO_2$  is regenerated.



Figure 27: CO<sub>2</sub> loading in the lean and rich solutions.



Figure 28: Ammonia in clean syngas from the absorber.

As explained earlier, the bubble cap trays did not perform as designed. This resulted in operating the absorber with only two stages. Ammonia emissions from absorber were recorded using an FTIR spectrometer. Figure 28 shows ammonia in clean syngas stream from absorber. This gas stream was subsequently passed through a water-wash column. Ammonia in clean syngas from the water-wash column was monitored using Draeger tubes, and it was found to be 250 ppm or less.

Process modeling using ASPEN shows that it is possible to reduce ammonia emissions to < 50 ppm using a water wash with well-designed bubble cap trays. In a future test campaign, we expect to install commercially designed bubble cap trays and show efficacy of ammonia capture in the water-wash column. Ammonia from the water-wash waste stream will be recovered in a sour-water stripper in a commercial plant.

Electric heat tracing was used to maintain the 120°F temperature in the bottom of the absorber to prevent precipitation from the rich solution. All rich-solution transfer lines from the absorber to the regenerator were heat traced. The second stage of the absorber had a recirculation loop to cool the semi-rich solution. Fresh lean solution was added to the second absorber stage to reduce ammonia emission.



Figure 29: Absorber bottom-stage temperature profile.

Both the absorber and regenerator were operated at similar pressures (~ 350 psig) to reduce pumping needs across pressure boundaries. High-pressure absorption results in reduced ammonia emission and thus lowers the operating cost by minimizing solvent make-up costs.



Figure 30: Absorber and regenerator pressure.



Figure 31: Reboiler temperature for thermal siphon.

A vertical reboiler was used to regenerate the rich solution using steam in a countercurrent heat exchanger at moderate pressure. The temperature in the regenerator was maintained by controlling the steam flow in the reboiler. It was determined that both  $CO_2$  and  $H_2S$  can be regenerated in a single vessel, and the lean solution is recirculated back to the absorber. In a commercial system, the heat source in the reboiler can be syngas and this can potentially result in significantly increased efficiency and reduced operating expenses. It may be noted that ammonia does not degrade at the operating temperature of reboiler. The higher the reboiler temperature, the higher the pressure of regenerated streams of  $CO_2$  and  $H_2S$ —thereby reducing the compression needs of  $CO_2$  stream after converting  $H_2S$  to elemental sulfur in the BPSC.

The regenerated stream was tested for composition. Grab samples from the regenerated gas stream were taken in a pressure vessel and analyzed with a GC. It is evident that the solubilities of fuel species like  $H_2$ , CO, and  $CH_4$  in solvent are very low; thus, there is minimal loss of fuel species.



Figure 32: Regenerated gas stream main constituents.

The solubilities of methane and carbon monoxide were below the detection limit of the GC. Figure 33 shows the GC measurement results for  $CH_4$  and CO in the regenerated gas stream, and Figure 34 shows the GC measurement results of hydrogen and argon in the regenerated gas stream.



Figure 33: Methane and carbon monoxide in the regenerated stream.



Figure 34: Hydrogen and argon in the regenerated gas stream.

Coriolis meters were used to measure various gas flows in the process system, including inlet raw syngas flow, clean syngas return flow, and regenerated gas stream flow. Figure 35 shows a mass balance between product and inlet gas flow rates.



Figure 35: Gas mass balance.

The AC-ABC / BPSC system setup at the NCCC is rated for ~ 150,000 scfd syngas flowrate. The absorber and regenerator columns are 8" ID and 40-feet tall. This is a relatively small system to measure heat of reaction. ASPEN Plus was used to model the system and to calculate the heat of reaction. Based on the modelling results, the program predicted the heat of reaction for  $CO_2$  and  $H_2S$  absorption and regeneration as ~ 690 BTU/l b or 1.45 GJ/ton of regenerated gas stream.

The measured heat input was higher than calculated due to heat losses in a small system. Figure 36 shows the heat of reaction based on steam input to the system during the 7-day steadystate run.



Figure 36: Energy input to reboiler.

#### Corrosion

During the second test campaign, some dark solids were observed in the solvent. Four samples were collected and sent for analysis to an outside lab. X-ray diffraction (XRD) analysis showed iron and sulfides.

The waste collected during the test campaign included spent solvent, rinse water, and stream from two water-wash columns. As per the report provided by the NCCC, the spent solvent contained 28 mg/kg of chromium (the specification for disposal is 5), the rinse water had 3.6 mg/kg, and the waste water from water-wash columns contained 0.154 mg/kg.

The above tests indicate possible corrosion in the system; however, it is not yet clear where this corrosion may be happening. The regenerator and reboiler are the hottest parts of the system, but the solvent is lean in H<sub>2</sub>S and CO<sub>2</sub> in these vessels and rich in ammonia. Another possibility is in the bottom of absorber, where rich solution is circulated at  $120 - 140^{\circ}$ F. According to one study of sour-water strippers in refineries, SS316 is more prone to corrosion than carbon steel, especially in ammonia-rich environments due to ammonia-metal complex formation.

During the first test campaign in August/September 2015, water samples were collected at the beginning and end of each of five runs. Some chromium was reported in the water samples of the first two of the five test runs during the test campaign. Solids transported with syngas during the first two test runs (as shown by presence of calcium and magnesium) were assumed to be the sources of chromium during the first test campaign. There was no chromium recorded in test Runs 3, 4, and 5 of the first test campaign.

The location of corrosion is not clear, and we plan to use corrosion test coupons in multiple locations during the next test campaign

#### H<sub>2</sub>S to Sulfur

The regenerated gas stream, CO<sub>2</sub>, and H<sub>2</sub>S from the AC-ABC system are fed to the Bechtel designed BPSC skid at 350 psig, and SO<sub>2</sub> is injected into the feed stream in a stoichiometric ratio. The BPSC has three catalytic beds that operate in a batch mode as follows: lead-lag-regenerate. At this small scale of operation, there were some problems with achieving steady SO<sub>2</sub> flow. SO<sub>2</sub> is stored at site as liquid in cylinders and is pumped in a loop using a diaphragm pump to 450 psig. A Coriolis-based mass-flow controller directs a controlled flow to the BPSC system through a vaporizer. SO<sub>2</sub> liquid vaporizes in the vaporizer and mixes with the feed gas from the AC-ABC system in a catalytic bed reactor, where sulfur is formed. The catalytic beds are regenerated periodically to collect sulfur that would normally flow in a liquid state to a collection vessel. Due to heat-tracing problems, liquid sulfur did not flow as expected.

Overall, the pilot-plant operation showed that the BPSC technology is capable of converting > 99.5 vol%  $H_2S$  gas to elemental sulfur with  $H_2S$  in the product gas at ppm levels. Sulfur formed on the catalyst at sub-dewpoint conditions according to the thermodynamic concept during the absorption step. Although only eight regenerations were completed and no long-term aging data was collected, the unit recovered the sulfur as a high-purity liquid. The limited data collected indicates that the parameters used in the sulfur condenser design are sufficient to move forward to the next design phase, where they can be optimized further.

Figure 37 shows the catalyst bed temperatures during a regeneration sequence. Although steady, the difference in temperatures along the depth of the bed at steady-state reflect problems encountered with temperature control. The variances after time 1977 at the end of the regeneration, reflect the catalyst wall temperature dependence upon the setting of the electric tracing and the catalyst temperature dependence upon flow.



Figure 37: BPSC regeneration temperature chart.

Figure 38 shows the outlet plenum of a sulfur condenser. Due to heat tracing issues, the recovered sulfur did not flow into the sulfur flash drum, but it was found after shutdown to have successfully regenerated from the catalyst, entered the regeneration cooler, and condensed in the appropriate location. Thus, the underlying concept has been proven. The bright yellow color of the sulfur indicates a very high-purity sulfur product, probably in the 99.9+ percent range.

# ILLUSTRATIONS



**Figure 38**: The outlet plenum of the BPSC regeneration cooler (sulfur condenser) – close-up view after the second test campaign.

![](_page_46_Picture_5.jpeg)

**Figure 39**: The outlet plenum of the BPSC regeneration cooler after the second test campaign (sulfur condenser).

![](_page_47_Picture_2.jpeg)

Figure 40: A photograph of the AC-ABC pilot plant at the NCCC.

![](_page_47_Picture_4.jpeg)

Figure 41: A photograph of the process skids.

![](_page_48_Picture_2.jpeg)

Figure 42: A photograph of the sulfur reactors.

![](_page_48_Picture_4.jpeg)

Figure 43: A photograph of skid B-1 with the sulfur condensers.

![](_page_49_Picture_2.jpeg)

Figure 44: A photograph of the electrical and control panels.

#### **TECHNO-ECONOMIC ANALYSIS**

Tables 2 and 3 summarize the costs of CO<sub>2</sub> capture from coal-fired power plants using the AC-ABC process. Case B5A from National Energy Technology Laboratory (NETL) "*Cost and Performance Baseline for Fossil Energy Plants, Volume 1b: Bituminous Coal (IGCC) to Electricity,*" *Revision 2b*, DOE/NETL, July 2015, a non-capture IGCC plant with a GE gasifier, is used as the reference case. The CO<sub>2</sub> capture costs using the AC-ABC process are compared to the costs of using a dual-stage Selexol process (Case B5B). CO<sub>2</sub> capture costs are 25-32% lower using the AC-ABC process than the Selexol process, and the capital costs required to implement the AC-ABC process are 48% lower than the Selexol capital costs.

Powe	r Plant Inputs	DOE Case B5A: Baseline GEE IGCC- No Capture	DOE Case B5B: Selexol Capture	AC-ABC CO <sub>2</sub> & H <sub>2</sub> S Capture
Heat	Rate (BTU/kWh)	8,756	10,458	10,299
Gross	Power, kWe	747,800	734,000	714,173
Net P	ower, kWe	622,050	543,250	551,634
Total	Overnight Cost (\$/net-kW)	\$3,036	\$4,195	\$3,701
ΪţΥ	Net Power (GWh/year)	5,449	4,759	4,832
Capac Factor	CO <sub>2</sub> Captured (million metric tons per year)	-	4.0	4.0
Cost	of Electricity (COE)			
	Power Plant Capital	53.7	74.3	65.6
۷h)	Power Plant Fuel	25.8	30.8	30.3
ξ	Power Plant Overhead and Maintenance	23.1	30.5	28.7
(\$)	CO <sub>2</sub> Transport, Storage, and Monitoring	-	9.2	9.2
COE	Total	102.6	144.8	133.7
	COE (% Increase)		41.2%	30.4%
Avoid	led Cost (cost of CO <sub>2</sub> capture, \$/metric ton)		\$50.21	\$37.89
Cost	of CO <sub>2</sub> Captured, \$/metric ton		\$39.26	\$26.71

Table 2: CO<sub>2</sub> capture costs compared to the IGCC base case (Case B5A).

Capital Cost, 2011	DOE Case B5A: Baseline	DOE Case B5B:	AC-ABC $CO_2$ &
Selexol		\$251,145	
SRI AGR			\$121,104
CO <sub>2</sub> Compression		\$84,088	\$17,212
Water-Gas Shift Reactors		\$21,370	\$17,981
Claus		\$40,699	
Pressure Swing Claus			\$49,615
Subtotal (WGS, AGR, H <sub>2</sub> S Conversion, and CO <sub>2</sub> Compression steps only)		\$397,302	\$205,912
Total Overnight Cost (TOC) of Complete IGCC Plant	\$1,888,393	\$2,278,752	\$2,041,739

Table 3: Capital cost comparison to the IGCC base case (Case B5A).

#### CONCLUSION

Based on the test results from the two test campaigns and the techno-economic analysis results, the AC-ABC technology is able to meet DOE's performance goal of 90% CO<sub>2</sub> capture with a cost of  $CO_2$  capture that is much lower than the DOE goal of \$40/ton of  $CO_2$  and with significant system availability even at the small pilot-scale level. Solvent-based technologies have proven themselves to be effective and relatively easy to operate compared to some of the newer technologies for  $CO_2$  capture. The equipment for solvent technologies, i.e., pumps, valves, heat exchangers, etc., are readily available and the scale-up factors and challenges are well understood in the industry. We do not envision any new custom-built equipment, which will increase the likelihood of success at larger-scale demonstration level. System modeling tools, property methods, and solvent performance data are well developed and documented and are readily available and require fewer assumptions and extrapolations. The ammoniated solution used in this process is readily available. The low cost of the solvent coupled with demonstrated low losses from the process significantly reduce the operating costs of the plant. There is a vast industrial experience in ammonia-based processes, including operation of sour-water strippers across refineries and other industries. Our long-term goal is to sequester and/or utilize CO<sub>2</sub> instead of venting it to the atmosphere; therefore, the availability of high-pressure CO<sub>2</sub> from this process offers a unique benefit over the currently available technologies. Although the commercial interest in IGCC for power generation has declined over the past few years, there is a potential for this technology to be used for other syngas applications such as the production of ammonia and other chemicals, for acid gas removal from biomass gasification, and for acid gas removal from natural gas.