NO_x Control Options and Integration for US Coal Fired Boilers

Quarterly Progress Report

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Abstract

This is the tenth Quarterly Technical Report for DOE Cooperative Agreement No: DE-FC26-00NT40753. The goal of the project is to develop cost effective analysis tools and techniques for demonstrating and evaluating low NO_x control strategies and their possible impact on boiler performance for firing US coals. The Electric Power Research Institute (EPRI) is providing co-funding for this program. This program contains multiple tasks and good progress is being made on all fronts.

During this quarter, progress was made on the computational simulation of a full-scale boiler with the purpose of understanding the potential impacts of burner operating conditions on soot and NO_x generation. Sulfation tests on both the titania support and vanadia/titania catalysts were completed using BYU's *in situ* spectroscopy reactor this quarter. These experiments focus on the extent to which vanadia and titania sulfate in an SO₂-laden, moist environment. Construction of the CCS reactor system is essentially complete and the control hardware and software are largely in place. A large batch of vanadia/titania catalyst in powder form has been prepared for use in poisoning tests. During this quarter, minor modifications were made to the multi-catalyst slipstream reactor and to the control system. The slipstream reactor was installed at AEP's Rockport plant at the end of November 2002. In this report, we describe the reactor system, particularly the control system, which was created by REI specifically for the reactor, as well as the installation at Rockport.

Table of Contents

DISCLAIMER	i
ABSTRACT.....	ii
TABLE OF CONTENTS	iii
EXECUTIVE SUMMARY	1
EXPERIMENTAL METHODS	3
Task 1 Program Management	3
Task 3 Minimization of Impacts	5
Task 4 SCR Catalyst Testing.....	10
RESULTS AND DISCUSSION	33
CONCLUSIONS	34
LITERATURE REFERENCES	35
APPENDIX: SLIPSTREAM REACTOR OPERATING PROCEDURES	

Executive Summary

The work to be conducted in this project received funding from the Department of Energy under Cooperative Agreement No: DE-FC26-00NT40753. This project has a period of performance that started February 14, 2000 and continues through September 30, 2003.

Our program contains five major technical tasks:

- evaluation of Rich Reagent Injection (RRI) for in-furnace NO_x control
- demonstration of RRI technologies in utility boiler scale field tests
- impacts of combustion modifications (including corrosion and soot)
- ammonia adsorption / removal from fly ash
- SCR catalyst testing

To date good progress is being made on the overall program. We have seen considerable interest from industry in the program due to our initial successful field tests of the RRI technology and the corrosion monitor.

During the last three months, our accomplishments include the following:

- The results of full-scale application of the soot-formation model integrated into a CFD code illustrate that the limited mixing in the system can cause locally oxygen deficient zones resulting in inefficient destruction of soot even at high temperature (> 1500 K) and potential soot emission in flue gas. The impact of burner stoichiometry can be significant on soot formation/destruction as well as on NO_x emission. The level of detail provided by the CFD simulations can be a valuable aid in understanding the mechanisms by which combustion modification affects soot formation/destruction and NO_x emission.
- During this quarter, in situ analyses of vanadium-based SCR catalyst systems were completed. These are preliminary data primarily intended as shakedown for the system. Results to date indicate that the system produces results that represent improvements compared to literature examples of similar experiments (but for different catalyst systems). Investigations of surface chemistry specific to vanadium-based SCR catalysts show mixed results. We have as yet been unable to reproduce surface sulfation described by some authors in the literature. However, we have reproduced substantial ammonia and NO adsorption data that are consistent with literature sources.
- Construction of the CCS reactor system is essentially complete and the control hardware and software are largely in place. A problem encountered with ammonia analyzer interferences is close to being resolved.
- A large batch of vanadia/titania catalyst in powder form has been prepared for use in poisoning tests. An experimental design for poisoning of this catalyst with calcium and sodium minerals over a range of concentrations has been formulated.
- Minor modifications were made to the slipstream reactor and to the control system. The slipstream reactor was installed at AEP's Rockport plant at the end of November 2002. An operating manual for the reactor was written and given to the plant. Details of ammonia handling and safety training remain to be completed; REI is working

with the plant to complete these. AEP must also review and sign a Host Site Agreement before the testing can begin. In parallel, REI is negotiating with DOE the contract for the mercury oxidation tests to be carried out on the slipstream reactor.

Experimental Methods

Within this section we present in order, brief discussions on the different tasks that are contained within this program. For simplicity, the discussion items are presented in the order of the Tasks as outlined in our original proposal.

Task 1 - Program Management

During October 31 – November 1, 2002 REI hosted a visit by the DOE COR for this project. A project review meeting was held at the REI offices which provided the COR with an update on the methodology, accomplishments, remaining work, commercialization (technology transfer) status and potential future work for each Task within this project. In addition, as part of the project review meeting, the EPRI Program Manager for NO_x Control provided an overview of the current status and future needs for NO_x control, from the perspective of the electric utility industry. In addition to the project review meeting held at the REI offices, site visits to the University of Utah and Brigham Young University were conducted to allow the COR to meet with project team members from these institutions in a one-on-one setting and to see first hand their respective facilities.

The slipstream reactor designed to test the deactivation of SCR catalysts in the field has been installed at the AEP Rockport plant. Due to an outage on the boiler, it is anticipated that data collection will not commence until at least January, 2003. Further details are provided below in the section describing the accomplishments for Task 4.

Industry Involvement

Fuel-Tech and RJM have issued official news announcements highlighting the first commercial installation of the Rich Reagent Injection (RRI) NO_x control technology. Further details are available on the Fuel-Tech web page (http://www.fueltechnv.com/press_frameset.htm) under the November 5, 2002 news announcement and on the RJM web site in the news announcement section (<http://www.rjm.com/pdf/Exelon.pdf>).

Results from portions of this research program have been reported to industry through technical presentations at recent conferences and publications submitted to archival journals:

- A podium presentation entitled “Modeling and Demonstration of Rich Reagent Injection for NO_x Control in AmerenUE's Sioux Unit 1” was provided at the 2002 Power-Gen International, December 10-12, 2002, Orlando, Florida that highlighted modeling and field tests of RRI in the Ameren Sioux Unit #1 boiler that demonstrated RRI can provide NO_x reductions of 30% from full load baseline emissions with OFA of approximately 0.4 lb NO_x/MMBTU with no ammonia slip [Cremer et al, 2002].
- Two papers that highlight our progress on evaluating a corrosion probe for real time monitoring of waterwall wastage have been submitted to archival journals.

- A paper entitled “On-Line Technique for Corrosion Characterization in Utility Boilers” has been accepted for publication in *Fuel* [Linjewile et al., 2002].
 - A second paper, entitled “Prediction and Real-time Monitoring Techniques for Corrosion Characterization in Furnaces”, has been submitted to *Materials at High Temperatures* [Davis et al., 2002].
- An abstract for a paper entitled “Modeling The Impacts Of Soot From Low-NO_x Combustion Systems” that highlights our efforts on developing a CFD based model for soot generation and destruction for coal combustion has been accepted for presentation at the 28th International Technical Conference on Coal Utilization & Fuel Systems, to be held March 10-13, 2003 in Clearwater, Florida, USA [Shim et al, 2003a]. The paper includes a comparison of predicted and measured values for soot in a pilot scale test furnace and a study of the impact of including soot in simulations of a full scale coal fired utility boiler that uses a low NO_x firing system. An abbreviated version of the material in the paper will also be presented at the upcoming ACERC conference to be held at Brigham Young University [Shim et al, 2003b].
- A podium presentation entitled "Networked Control System for a Catalyst Deactivation Test Reactor" was presented to the National Instruments NIWeek held in Austin, Texas, August 14-16, 2002 that highlighted several novel techniques used by REI software engineers in the development of control system software for the SCR slip stream test reactor. [Swensen et al, 2002]

Task 3 – Minimization of Impacts

Task 3.2 Soot

During this quarter, progress was made on the computational simulation of a full-scale boiler with the purpose of understanding the potential impacts of burner operating conditions on soot and NO_x generation.

The boiler simulated is an 800 MW front- and rear-wall-fired unit with 16 single register B&W low NO_x burners in four elevations and four columns at each wall and an overfire air system. Computational mesh generated for the unit is shown in Figure 1. Top row burners at each wall are out of service. 6 NO_x ports at the front wall and 5 at the rear wall are used to introduce staged air at the same elevation. The exit of the model was at the horizontal nose plane of the furnace. Three levels of air staging were simulated with a fixed overall stoichiometry of 1.165 and a fixed firing rate of 7,800 MMBtu/hr to evaluate the impacts of staging level on soot formation. The burner stoichiometric ratios and air splits of each parametric case are shown in Table 1. In Param1, the burner was staged deeply using a burner stoichiometric ratio of 0.7. In Param2, burner stoichiometry was increased from 1.067 of Base to 1.118 and NO_x ports were not used for air injection. Though soot measurements are not available for the unit, the boiler operators have reported soot problems in the stack with Base operating condition.

Table 1. Operating conditions for the full-scale CFD simulations.

	Base	Param1	Param2
Overall SR*	1.165	1.165	1.165
Burner SR	1.067	0.7	1.118
<i>Air distribution:</i>			
BOOS [#]	4.0%	4.0%	4.0%
NO _x ports	4.4%	35.9%	0.0%
Burner	91.6%	60.1%	96.0%

* SR = stoichiometric ratio

BOOS = Burner Out Of Service, top row burner

The standard fuel fired in this unit is a blend of McElory and Cumberland bituminous coals with a grind of 99% through 50 mesh and 75% through 200 mesh. Table 2 summarizes the properties of these fuels.

Table 2. Fuel properties for coal as used in the full-scale CFD simulations.

Ultimate Analysis		Proximate Analysis	
C	67.3%	Volatiles	30.8%
H	4.2%	Fixed Carbon	50.6%
O	5.6%	Moisture	7.0%
N	0.8%	Ash	11.6%
S	3.6%	HHV (Btu/lb)	12158.8
Ash	11.6%		
H ₂ O	7.0%

Figure 1. Computational mesh generated for an 800 MW pulverized coal-fired unit

Table 3 summarizes the full-scale CFD simulation results. The effects of air staging were generally positive. As the burner was staged more deeply in Param1, the NO_x emission was reduced 34 % reduction from Base. In Param2, with no air staging, the NO_x increased by 9 % from Base. In Param1, CO and LOI were also reduced by 68 % and 17 %, respectively. In Param2, CO and LOI decreased by 35 % and 35 %, respectively. Figure 2 illustrates NO_x generation rate as a function of the distance from the bottom of hopper for three cases. Deeply staged Param1 showed lowest the NO_x generation rate. The case with no air staging (Param2) showed the highest NO_x generation rate, resulting in high NO_x concentration at the exit.

Table 3. Full-scale CFD simulation results

	Base	Param1	Param2
NO_x (ppm at 3% O_2 dry)	336	221	367
LOI (%)	7.9	6.6	5.1
CO (ppm, dry)	3,695	1,170	2,400
Exit Temperature (K)	1,516	1,561	1,513

Figure 2. NO_x, lb/hr as a function of distance from the bottom of hopper for three cases.

Figure 3 shows the average soot volume fraction and equivalence ratio as a function of the distance from the bottom of hopper. High soot concentration occurs at fuel rich conditions (equivalence ratio > 1.0) near the burners. The model shows that even at the stoichiometric or fuel-lean condition (overall), soot particles are not fully oxidized or destroyed and those soot particles will end up in flue gas. This can be explained by a limited mixing between fuel and air and resulting oxygen deficient condition in the system, which prevents soot destruction and facilitates soot growth/agglomeration. Figure 4 shows the predicted average soot volume fraction for three cases as a function of distance from the bottom of hopper. The figure shows the general trend that the deeper the staging, the more soot is formed. Additional air introduction in Base and Param1 beyond the firing zone and overall fuel-lean condition in Param2 were not enough to burn out soot particles due to limited mixing. Predicted average soot particle sizes at the exit were also increased as shown in Figure 4. The average soot particle size in Param1 is over 1 μm in diameter possibly due to long residence time under fuel-rich condition resulting in extensive soot growth and agglomeration (the model does not distinguish between single soot particles and agglomerated particles). Figure 5 illustrates the profiles of soot volume fraction and oxygen concentration at the exit plane of the furnace. It can be clearly seen that the local oxygen deficiency prohibits complete burn out of soot particles even at high temperature (exit temperature for all three cases > 1500 K). In addition, the decreasing gas temperature beyond the nose of the furnace and at the heat transfer sections will make it difficult to achieve further oxidation of soot particles. Therefore, those soot particles at the nose plane shown in Figure 4 and 5 have a high potential to remain in flue gas.

Figure 3. Soot volume fraction and equivalence ratio as a function of distance from the bottom of the hopper for Base.

Figure 4. Soot volume fraction as a function of distance from the bottom of hopper for three cases. Predicted soot particle diameters at the exit are also shown.

Figure 5. Soot volume fraction and oxygen concentration at the exit plane for Base, Param 1, and Param 2.

Task 4 - SCR Catalyst Testing

The purpose of this task is to perform a combination of basic and applied R&D, with heavy focus on laboratory and field tests, to develop a better understanding of the “real” costs associated with using selective catalytic reduction (SCR) for coal-fired boilers using US coals and a coal/biomass blend. Within this task there are four principal sub-tasks:

Task 4.1: Technology assessment on fundamental analysis of chemical poisoning of SCR catalysts by alkali and alkaline earth materials

Task 4.2: Evaluation of commercial catalysts in a continuous flow system that simulates commercial operation

Task 4.3: Evaluation of the effectiveness of catalyst regeneration

Task 4.4: Develop a model of deactivation of SCR catalysts suitable for use in a CFD code

Sub-tasks 1 and 3 are being principally performed at Brigham Young University under the direction of Professors Larry Baxter and Calvin Bartholomew. The work effort for sub-tasks 2 and 4 is being performed by REI, under the supervision of Dr. Constance Senior, with assistance from the University of Utah (Professor Eric Eddings and Dr. Kevin Whitty) on sub-task 2.

Task 4.1 Technology Assessment

The objectives of this subtask are (1) to supplement the largely complete SCR-catalyst-deactivation literature with results from new laboratory-scale, experimental investigations conducted under well-controlled and commercially relevant conditions, and (2) to provide a laboratory-based catalyst test reactor useful for characterization and analysis of SCR deactivation suitable for samples from commercial facilities, slipstream reactors, and laboratory experiments. Two catalyst flow reactors and several additional characterization systems provide the analytical tools required to achieve these objectives. The flow reactors include the *in situ* surface spectroscopy reactor (ISSR) and the catalyst characterization system (CCS), both of which were described in more detail in the Technical Quarterly Report for July through September 2002. The ancillary characterization systems include a temperature-programmable surface area and pore size distribution analyzer, scanning electron microscopes and microprobes, and catalyst preparation systems.

The sample test matrix includes two classes of catalysts: commercial, vendor-supplied SCR catalysts and BYU-manufactured, research catalysts. The commercial catalysts provide immediate relevance to practical application while the research catalyst provides less fettered ability to publish details of catalyst properties. The five commercial catalysts selected for use come from most commercially significant catalyst manufacturers (Cormetech, Haldor Topsoe, Hitachi, and Siemens) and provide a wide range of catalyst designs and compositions. The in-house catalyst allows detailed analysis and publication of results that may be more difficult with the commercial systems. This catalyst suite provides a comprehensive test and analysis platform from which to determine rates and mechanisms of catalyst deactivation. The result of this task will be a mathematical model capable of describing rates and mechanisms of deactivation.

ISSR Overview

The purpose of the FTIR-ISSR is to provide definitive indication of surface-active species through in situ monitoring of infrared spectra from catalytic surfaces exposed to a variety of laboratory and field conditions. The ISSR provides in situ transmission FTIR spectrometer measurements of SO₂, NH₃, and NO_x, among other species. Absorption and desorption behaviors of these and other species are monitored. Quantitative indications of critical parameters, including Brønsted and Lewis acidities on fresh and exposed catalysts will be included. Indications of coadsorption of NH₃ and NO_x will help elucidate mechanisms and rates of both reactions and deactivation.

FTIR Study of Vanadia Catalyst Sulfation

Sulfation tests of six titania support and vanadia/titania catalysts were completed this quarter. In addition, repairs and upgrades to the existing equipment were completed, including addition of a new computer and data acquisition system for the FTIR spectrometer, a dry-air filter for the FTIR system, and two mass flow controllers for the reactor.

A series of twenty-four hour sulfation experiments on titania, a 2% vanadia on titania catalyst, and a 5% vanadia on titania catalyst provided data that are largely consistent with literature data. These experiments focused on the extent to which vanadia and titania are sulfated in an SO₂-laden, moist environment. All experiments included oxidizing, SO₂-laden environments (nominal 5% oxygen and 0.23% SO₂) and varied from dry to moist (nominal 2.5% moisture) flows. All samples were pre-oxidized in SO₂-free flows. Details of these sulfation experiments are listed in Table 4.

Table 4. Summary of Experimental Conditions

Sample	Sample	Notes	Gas Stream Composition, %						Flow rate scm	Temp °C	Windows	XPS
Name			He	O ₂	SO ₂	NH ₃	NO	H ₂ O				sample
V8TH	5% V ₂ O ₅ /TiO ₂	Preoxidation	95	5				0	49.75	380	KCl+CaF ₂	
	24hrs	Sulfation	94.95	4.76	0.29			2.4	52.5	380	KCl+CaF ₂	Pellet
V8TO	5% V ₂ O ₅ /TiO ₂	Preoxidation	95	5				0	52.5	366-380	KCl+CaF ₂	
	24hrs	Sulfation	94.48	5.29	0.23			0	39.69	380	KCl+CaF ₂	Pellet
V9TO	2% V ₂ O ₅ /TiO ₂	Preoxidation	90	10				0	50	377	KCl+CaF ₂	
	24hrs	Sulfation	94.06	5.29	0.23			2.4		370-390	KCl+CaF ₂	Pellet
V9TH	2% V ₂ O ₅ /TiO ₂	Preoxidation	90	10				0	50	377	KCl+CaF ₂	
	24hrs	Sulfation	92.6	4.9	0.23			2.2			KCl+CaF ₂	Pellet
TiO ₂ -H	Pure TiO ₂	Preoxidation	90	10				0	50	385	KCl+CaF ₂	
	24hrs	Sulfation	92.6	4.9	0.23			2.2	36.89	380	KCl+CaF ₂	Pellet
TiO ₂ -4	Pure TiO ₂	Preoxidation	90	10				0	50	385	KCl+CaF ₂	
	24hrs	Sulfation	94.06	5.69	0.23			0	36.89	380	KCl+CaF ₂	Pellet

Both in situ FTIR spectroscopy analyses and XPS analyses provided data regarding the extent of surface sulfation, as summarized in Table 5

Sulfation of pure TiO_2 with a moist, SO_2 -laden gas under conditions typical of SCR operation provides a baseline comparison for experiments involving sulfation of vanadia catalysts. Based on *in situ*, real-time FTIR analyses steady-state sulfation was reached in about 5 h at conditions of 35-50 sccm, 370-390°C, 0.2-0.3% SO_2 . Nevertheless, sulfation experiments were continued for an additional 19 hours (a total of 24 h), since literature data for similar catalysts and conditions indicate that 18-24 h may be required for complete sulfation.

Table 5. XPS analysis results

Sample		Elements	B.E.	% Atom
V8TO	5% V Sulfation without water	C1s	280.623	24.2
		O1s	529.609	56.0
		S2p	164.081	1.5
		Ti2p	453.81	16.5
		V 2p3/2	512.347	3.3
V8TH	5% V Sulfation with water	C1s	281.215	5.3
		O1s	529.601	65.8
		S2p	164.762	2.8
		Ti2p	454.354	20.2
		V 2p3/2	512.892	3.3
V9TO	2% V Sulfation without water	C1s	281.252	8.3
		O1s	529.99	67.7
		S2p	165.225	2.1
		Ti2p	455.909	20.9
		V 2p3/2	512.614	1.9
V9TH	2% V Sulfation with water	C1s	285.23	10.1
		O1s	530.86	65.2
		S2p	169.48	2.4
		Ti2p	458.95	20.2
		V 2p3/2	517.47	2.1
TiO ₂ -4	Titania Sulfation Without Water	C1s	280.485	8.7
		O1s	524.961	66.4
		S2p	163.95	2.8
		Ti2p	457.741	23.2
TiO ₂ -H	Titania Sulfation With Water	C1s	280	10.2
		O1s	525.48	70.7
		S2p	164.5	3.4
		Ti2p	458.95	15.6

Sulfation of pure TiO_2 with a moist, SO_2 -laden gas under conditions typical of SCR operation provides a baseline comparison for experiments involving sulfation of vanadia catalysts. Based on *in situ*, real-time FTIR analyses steady-state sulfation was reached in about 5 h at conditions of 35-50 sccm, 370-390°C, 0.2-0.3% SO_2 . Nevertheless, sulfation experiments were continued for an additional 19 hours (a total of 24 h), since literature data for similar catalysts and conditions indicate that 18-24 h may be required for complete sulfation.

IR spectra of the sulfated catalysts include one major sulfate-related feature with a doublet located near 1375 cm^{-1} (see Figure 6). It is unclear if these two peaks represent two different sulfate complexes or two different vibrational/rotational modes for the same complex. Moreover, the peak positions shifted to higher frequencies with increasing time during the sulfation test, indicating increasing sulfate acidity with time/surface coverage.

Figure 6. Results from 24-hour exposure of titania to an SO_2 -laden, moist gas.

It was observed that the intensity of the sulfate doublet decreased with increasing vanadia content for the sulfation tests that included water vapor. These results are consistent with those observed following treatment in dry gases (see previous quarterly report), i.e., the feature at $1370\text{--}1375\text{ cm}^{-1}$ in the FTIR spectra,

which is designated as an S=O stretching of sulfate species on catalysts surface, decreased dramatically with increasing vanadia content on the catalyst surface. This peak, in fact, was apparently not present on 5% vanadia catalyst during and after the sulfation moist test.

Others have observed the same trend of decreasing FTIR intensity with increasing vanadia concentration [Dunn et al., 1998; Jung and Grange, 2002; Amiridis et al., 1996]. Jung suggested that the S=O structure is prerequisite to the generation of acidic sites on sulfate promoted oxide samples and that a decreasing S=O surface concentration with increasing sulfur content on the surface could be due to the generation of polynuclear sulfate species at the expense of isolated sulfate species. However, our results indicate that S=O concentration from FTIR spectra is proportional to sulfur concentrations measured by XPS. Moreover, no other sulfur forms are detected by IR spectroscopy in our experiments.

Both Dunn and Amiridis indicate that formation of sulfate species on the surface of vanadia catalysts is not favored. Dunn observed that the surface concentration of sulfate species, as reflected by the intensity of the 1373 cm^{-1} band, dramatically decreases with increasing coverage of the surface vanadia species and that the molecular structure of the surface vanadia species was only slightly perturbed by the presence of sulfur. They also concluded that surface sulfate species adsorb on the most basic support hydroxyl species. However, Choo concluded that surface vanadate species titrate both basic and neutral support hydroxyls forming a complete close-packed monolayer. Amiridis et al. speculated that their observed decrease in sulfate species with surface vanadia concentration might derive from a repulsive interaction between vanadyl and sulfate species. Moreover, their ICP analysis was consistent with a decrease in surface sulfate with increasing surface vanadia.

Results of XPS analysis for six samples after 24-hour exposure to dilute SO_2 (three in the absence and three in the presence of water vapor) are summarized in Table 5. The third column of the table identifies the element and its electronic orbital analyzed by XPS. The XPS technique measures composition of surface and near-surface layers on the exterior of catalyst particles. Therefore, the observed surface concentrations of vanadia on the 2% samples of 2.1 and 2.4% are approximately the same as the nominal bulk concentration (i.e., 2%), while surface vanadia concentrations in the 5% samples (3.3% in both cases) are less than in the bulk, although they are still higher than in the 2% vanadia samples.

Figure 7. Sulfur content on TiO_2 and $\text{V}_2\text{O}_5/\text{TiO}_2$ as determined by XPS.

Surface sulfur concentrations measured by XPS (coverages ranging from 1.5 to 3.3% of the exposed surface) are significant for all 6 samples. A general trend of linearly decreasing sulfur coverage with increasing vanadium content is apparent in Figure 7 for samples sulfated in the absence and presence of water vapor (with the exception of a slight upturn in sulfur content for the wet sample of highest vanadium content). The significantly higher surface sulfur contents for the samples sulfated in the presence of water vapor indicates that water apparently promotes accumulation of sulfur on both the titania support and vanadia catalyst.

Several authors have suggested that SO_2 or sulfate species react with catalyst surface by anchoring to surface hydroxyl groups, which may be the same surface sites to which vanadia species bind. For catalysts in which vanadate species reach a monolayer distribution, there are few surface hydroxyl groups remaining on the titania surface. The absence of an S=O peak in FTIR spectra for the 5% vanadia catalyst is not consistent with the XPS analyses indicating a significant concentration of sulfur to be present on the catalyst surface. Accordingly, we conclude that sulfate species either adsorb on surface hydroxyls on the titania surface and alter their infrared cross sections substantially or react with vanadia species to form a species not active in this portion of the infrared spectrum. As the change in the backscatter energies (BE) of sulfur in the XPS analysis is insignificant, sulfur in all three samples should be in the same oxidation state (+6).

Although the 1375 cm^{-1} IR peak for vanadia catalysts is either very small or unobservable, suggesting that the content of S=O is very low, additional peaks at $1632\text{--}1633\text{ cm}^{-1}$ and $1124\text{--}1133\text{ cm}^{-1}$ are observed in the FTIR spectra for sulfated 2 and 5% vanadia catalysts after exposure to air (Figures 8 and 9). Since these new peaks do not appear on the pure titania samples, they may be caused by adsorption on vanadia-sulfate centers. The IR spectrum of vanadyl sulfate exhibits intense peaks from 1100 to 1150 cm^{-1} . Jung et al. indicate that IR spectra of sulfated metal oxides normally contain a strong adsorption band near $1380\text{--}1360\text{ cm}^{-1}$ and a broad band around $1000\text{--}1200\text{ cm}^{-1}$. Therefore, the sulfur species accounting for the residual sulfur could be a species similar to vanadyl sulfate that, for reasons we have yet to determine, is not infrared active under reaction conditions.

Figure 8. Infrared spectra of five percent vanadia on titania substrate after exposure to room air.

Figure 9. Infrared spectra of two percent vanadia on titania substrate after exposure to

CCS Overview

The catalyst characterization system (CCS) provides capabilities for long-term catalyst exposure tests required for ascertaining deactivation rates and mechanisms and a characterization facility for samples from the slipstream reactor to determine changes in reactivity and responses to well-controlled environments. This system, described in detail in our last report, simulates industrial flows by providing a test gas with the following compositions: NO, 0.10%; NH₃, 0.1%; SO₂, 0.1%; O₂, 2%; H₂O, 10%; and He, 87.7%. Both custom and commercial catalysts are tested as fresh samples and after a variety of laboratory and field exposures under both steady and transient conditions.

The purpose of the CCS is to quantitatively determine deactivation mechanisms by measuring specific, intrinsic catalyst reactivity of custom (laboratory) and commercial catalysts under a variety of conditions. These catalysts will be impregnated with a variety of contaminants, including Ca, Na, and K. In addition, the CCS will characterize samples of catalyst from slipstream field tests to determine similar data and changes in characteristics with exposure. Advanced surface and composition analyses will be used to determine composition, pore size distribution, surface area, and surface properties (acidity, extent of sulfation, etc.).

During this quarter construction and shake-down tests of the CCS reactor system was essentially completed and the control hardware and software are largely in place. A problem encountered with ammonia analyzer interferences is close to being resolved.

A large batch of vanadia/titania catalyst in powder form has been prepared for use in poisoning tests. An experimental design for poisoning of this catalyst with calcium and sodium minerals over a range of concentrations has been formulated. Helium functions as the carrier gas in this system since molecular nitrogen formed from NO_x is measured as part of the data analysis. NO_x derived nitrogen would represent an insignificantly small fraction of total nitrogen if nitrogen were used as a carrier gas. There is no indication in any literature of which we are aware that substitution of helium for nitrogen in any way alters rates or mechanisms of selective catalytic reduction of NO_x on vanadium-based catalysts.

Catalyst Preparation and Characterization

A $\text{VO}_x/\text{WO}_3/\text{TiO}_2$ catalyst was prepared for use in characterization and poisoning studies. The desired composition was 1 wt.% V and 9 wt.% W on TiO_2 . Enough catalyst was prepared in one batch to satisfy the needs of the entire experimental design in order to assure experimental consistency and precision. 150 grams of catalyst were prepared by incipient wetness according to the following procedure:

- 1) Mix TiO_2 with distilled water at a ratio of 1:1.75 by weight.
- 2) Dry at approximately 100°C for 24 hours.
- 3) Crush with mortar and pestle until fine powder is obtained.
- 4) Calcine at 600°C for 4 hrs to obtain a densified titania support.
- 5) Determine approximate pore volume for achieving incipient wetness by measuring the weight of water taken up by the dried support.
- 6) Make a solution of ammonium metavanadate, ammonium tungstate, and oxalic acid that will yield 1% V_2O_5 and 9% WO_3 on the titania support.
- 7) Add the solution prepared in step six to 135 grams of densified titania.
- 8) Dry the resulting paste at approximately 100°C for 24 hrs.
- 9) Calcine the dry catalyst at 550°C for 5 hrs.

A small amount of catalyst that had been prepared previously by an identical procedure was used to run an initial test to determine catalytic activity. The results of this test indicated a very active catalyst. This was a fresh catalyst that had not been sulfated and was in a gas stream containing 1000 ppm NH_3 , 1000 ppm NO , 2% O_2 , in helium at a space velocity of $15,000 \text{ hr}^{-1}$ and temperature of 350°C . More than 70% conversion of NO was observed for this catalyst at these conditions.

The three peaks at 0:00, 1:00 and 20:30 hours correspond to times when the feed was sampled to verify that the feed to the catalytic reactor contained 1000 ppm NO . The small step increase at the end of the run is the gas sample from a reactor that contained the same gas mixture as that noted above, with the addition of approximately 10% water vapor. This indicates that the activity of the catalyst prepared at BYU is higher in the presence than in the absence of water vapor. The cause of the bump in the data that occurred between times 1:30 and 5:00 is unknown.

Our experimental procedure calls for an NO conversion of only about 20%, considerably lower than that exhibited by our catalyst at these conditions. In order to decrease conversion the space velocity will be increased and temperature decreased, thus approximating a differential reactor.

NO Interference in NH_3 Measurement

NH_3 concentration data collected at the same time as those shown in Figure 10 are unexpectedly low, indicating that either a reaction occurs that consumes NH_3 and not NO, or NO is interfering with the NH_3 measurement. Figure 10 shows the NH_3 analyzer output (dark solid line) of about 700 ppm NH_3 when the flow calibration would predict 1000 ppm.

Figure 10. Initial catalytic activity test.

Two more tests were run to determine whether or not a side reaction of NH_3 was occurring or if NO was interfering with the NH_3 measurement. The first test involved running a gas mixture of about 900 ppm NH_3 , balance helium, through an empty reactor at room temperature and cycling the NO concentration from 0 ppm to 1000 ppm. These data suggest that the same reaction/interference occurs with or without the catalyst and at either elevated room temperature. To check for possible adsorption of NH_3 on the stainless steel tubing a second test was performed by bypassing the reactor system completely with plastic tubing and feeding a 1000 ppm NH_3 , 1000 ppm NO, balance helium gas stream directly to the gas analyzer. The sudden decrease in the NO measurement and increase in the NH_3 measurement at time 4:00 are a result of running out of NO.

Reactor System Control

A detailed description of the function of the SCR test reactor system and its LabVIEW™ control program has been given in previous quarterly reports. A few significant changes and improvements have been made since the last report (October 2002), which are summarized here. The reader is encouraged to note that, although there are still changes and improvements to be made, the control program is fully functional and can both control the various pieces of equipment as well as log gas composition data for all four reactors.

Feed Sampling The reactor system can analyze four powdered catalyst samples simultaneously, although only one of those samples can be analyzed at a time because there is only one set of gas analyzers. Furthermore, each reactor can be analyzed at one of two places: at the inlet before the catalyst (the “feed”) or at the outlet after the catalyst (the “effluent”). In its previous design, the control program sampled each of the four reactor effluents automatically at an interval specified by the operator, but did not have the capability to sample the reactor feed streams. The program has been modified so that there are two modes of valve operation: Auto or Manual. If the valve is placed on “Auto,” the program cycles through the four reactor effluent streams at intervals specified by the user (15 minutes is default). The time between data points may also be specified. A diagram of the controls for this Auto mode is shown in Figure 11.

Figure 11. Display of Multiposition Valve Automatic Function Control.

Conversely, if Manual operation is selected, the user may select any one of the ten total channels of the multiposition valve via a drop-down menu (Figure 12). In this way, any stream may be analyzed for an arbitrary amount of time, again at any sampling rate.

Figure 12. Display of Multiposition Valve Manual Function Control.

Mass Flow Controller Alarms The control program allows a user to specify the space velocity, gas concentrations, amount of catalyst used, etc. and will adjust gas flowrates accordingly. However, a negative result of these control capabilities is the potential to inadvertently set a flowrate that is beyond the maximum capability of a mass flow controller (MFC). If unchecked, this problem could affect the intended gas concentrations fed into the reactor and could damage mass flow controllers. A safeguard has been added to the program which will terminate the program and display a window that informs the user of this problem if too high a flowrate is set (Figure 13).

Figure 13. Pop-up window indicating an excessive setpoint.

In addition to the alarm described above, another common problem for which an alarm has been added is the case where a mass flow controller's actual flow deviates significantly from its

setpoint. This problem usually results from an operator error (i.e. forgetting to open a cylinder valve upstream or neglecting to replace an empty cylinder), although it has been known to occur in the case of at least one mass flow controller whose valve sometimes sticks. In order that a user may notice this problem at a quick glance, indicator lights have been added to the display in between the Setpoint and Actual flowrates displays (Figure 14), which show green if actual flow is within 5% (adjustable) of the setpoint, and show red otherwise.

Figure 14. Indicator lights showing significant deviations from MFC setpoint.

Several adjustments and corrections to the program have been made that are not apparent by interacting with the graphical-user interface, but are nonetheless important to the way the program operates. First, several items that were mismatched between the computer display and the actual reactor system have been fixed. For example, Reactor 2's actual pressure was being displayed under Reactor 4's pressure display.

Second, more information about which stream is being logged is available on the .txt files where the reactor data is stored. For example, Reactor 4's data file will indicate whether or not it is being sampled, as well as whether it is the feed or the effluent that is sampled. Prior to this version, the .txt file only indicated if the reactor was being sampled merely with a "yes" or a "no," whereas now it indicates either "feed," "exit," or "no." Similar and more-subtle changes that have been made to the code to make it more efficient are not summarized here.

Task 4.2 Evaluation of Commercial Catalysts for Power Plant Conditions

The objective of this task is to evaluate SCR costs at a deeper level and to improve estimates of actual costs. One of the prime motivations for this program is to look at the influence of the alkali and alkaline earth elements in biomass and how those affect SCR catalyst when biomass is co-fired with coal. This will be accomplished using a multi-catalyst slipstream reactor designed by the University of Utah. During this quarter, minor modifications were made to the reactor and to the control system. The slipstream reactor was installed at AEP's Rockport plant at the end of November 2002. In this report, we describe the reactor system, particularly the control system, which was created by REI specifically for the reactor, as well as the installation at Rockport.

Slipstream Reactor Description

The SCR reactor set up consists of the slip-stream reactor referred to as the SCR unit, a data acquisition and control enclosure (CONTROL BOX), an air supply controls enclosure (AIR BOX), an enclosure for sample conditioning and switching between lines (SEQUENCER) and a cabinet for housing the NO_x and O₂ continuous analyzers (CEMS). In addition the reactor is connected to an ammonia supply manifold and a suite of calibration gases. The complete system for the slipstream reactor consists of the equipment listed in Table 6. The following is a brief description of these components.

Figure 15 shows a schematic drawing of the reactor. There are six identical chambers. The overall flow through the system is controlled by a single eductor just upstream of the system exhaust. A venturi flowmeter at the inlet to the system provides feedback to a controller that adjusts a valve on the air line to the eductor. The eductor has a large flow range and turndown ratio. To ensure that the flow rates through the catalyst chambers are equal, the pressure drops are set equal by adjusting the flow of compressed air through eductors downstream of the catalysts. Air flow to the eductors flows through a set of six differential pressure control valves located in the AIR BOX.

Table 6. Components of SCR Slipstream Reactor

Quantity	Item
1	SCR Reactor
1	CONTROL BOX
1	AIR BOX
1	CEMs
1	SEQUENCER
4	Ammonia cylinders
3	Calibration gas cylinders
1	Local PC (located in office space)

Anhydrous ammonia will be injected into the flue gas stream, and blended with a static mixer, housed in a spool piece located above the catalyst chambers. The ammonia feed rate will be maintained by a mass flow controller, and will be adjusted based on the measured amount of NO_x entering the system. The molar ratio of NH₃/NO_x will be set at approximately 1.1 in order to ensure that NO_x destruction is not limited by stoichiometry and to give margin for swings in NO_x levels.

Figure 15. Schematic of SCR Slipstream Reactor

Six catalysts will be tested; four monolith and two plate, and will be configured as shown in Figure 16.

Figure 16. Arrangement of catalysts (plan view)

The four monolith catalysts will be installed in four sections each. Each section will have a cross section of 2.25 by 2.25 inches, and will be housed in a 48-inch long aluminum square tube with outer dimensions of 2.5 by 2.5 inches and 1/8" wall thickness. The overall cross section of each monolith catalyst will be 4.5 x 4.5 inches. The four tubes will be bunched together as a square with outer dimensions 5.0 x 5.0 inches.

The plate catalysts will be housed in square aluminum tubes with an inside dimension of 4.75 inches (5.0 inches outside, 1/8" wall thickness). Roughly 20 plates will be placed in the tube, resting in slots along opposite walls. This configuration will have the same outer dimensions as the groupings of monolith catalysts.

Catalyst samples will be taken three times during the testing period in order to diagnose deactivation. The total flow through the system will be adjusted downwards as catalyst samples are removed. The total cross sectional area of the two plate catalysts will remain the same throughout the test. Flow channels of the four monolith catalysts will be blocked off as catalyst samples are taken. The overall flow rate will be adjusted to correspond to the cross sectional area of the remaining catalyst chambers, and will follow the progression given below:

Initial configuration:	100.0%
After first sample has been removed:	83.3%
After second sample has been removed:	66.7%
After third sample has been removed:	50.0%

Samples of monolith catalyst will be taken by removing one of the four chambers entirely. The opening that remains will be blocked off at the inlet by a sloped cover to minimize ash deposition and flow disturbances.

Plate catalyst samples will be taken by removing every fourth plate, and replacing it with a non-reactive dummy plate.

The order of catalyst removal is shown in Figure 17. Blocked monolith chambers are shown by a gray square. Non-reactive dummy plates are shown in gray. Monolith chambers are removed starting from the outside in order to keep the profile as centered and uniform as possible.

The open cross section of the catalyst system decreases as samples are removed. To keep the flow velocity constant through the chambers, the overall flow through the system is decreased correspondingly as catalysts are removed, as described above in the section on the flow control system. In terms of the initial flow through the system, the overall flow follows the progression 100%, 83%, 67%, 50%. The pressure drops across the six catalyst sections (divided by catalyst type) are adjusted with butterfly valves so that they are identical to each other (as described above), so the flow velocity per catalyst chamber should be the same throughout the duration of the tests.

Figure 17. Catalyst removal plan.

sample line selection. Through an automatic valve-switching sequence set in the software, one of these gases at a time is sent to the CEM via heated stainless steel line. In the sequencer, provisions are also made of blowback of the sample lines. The CEMs consist of a Horiba Model CLA-510 Chemiluminescent NO-NO_x Analyzer and a Horiba Model MPA-510 Magnetopneumatic Paramagnetic O₂ Analyzer. In order to maximize accuracy, the pre-catalyst gas is analyzed before each post-catalyst measurement. Thus, analyses are performed in the order: pre, post 1, pre, post 2, pre, post 3, etc. The data from the analyses are logged and can be remotely accessed via modem or network. The control system was created specifically for this program and is described in more detail below.

The reactor flue gas intake will be attached to boiler at the economizer outlet where up to 250 scfm of gases will be withdrawn through a probe inserted in an existing port. The probe is also fitted with a thermocouple for monitoring the temperature of the flue gas as it exits the economizer. After passing through the reactor, the gases will be exhausted through an existing port at the air heater exit. The reactor itself is approximately 8 feet long, with a 25 x 30 inch footprint, and weighs approximately 1000 lbs. The reactor will be well insulated and securely fastened.

In order to minimize ash buildup in the system and maintain catalyst activity, large volumes of high-pressure air are periodically blown through the system. A line connected to the plant's compressed air system (~100 psig) runs to an automatic open/shut valve. This supplies air to a bank of "soot-blowing" nozzles placed above the catalyst chambers. When open, air is blown downwards into the catalyst chambers for about 10 seconds. The volumetric flow rate of this air is several times higher than that normally flowing through the system, and will blow out any ash that may have deposited. Prior to activating the "soot-blowing" sequence, the sampling system is isolated.

The system has seven sampling ports, one before the catalyst chambers and one after each of the six catalyst sections. The ports themselves consist of thin tubes that enter the channel and bend downwards, in line with gas flow. Each sampling port is fitted with a stainless steel frit for removal of particles laden in the sample. A sample drawn either from the SCR inlet or exit via a heated stainless steel line is sent to a sample-conditioning module housed in an enclosure called the SEQUENCER. The sequencer houses a sampling pump, a thermoelectric cooler for removal of moisture, candle filter and a bank of solenoid valves for

Anhydrous ammonia (stored in cylinders that hold approximately 150 lb each) is introduced into the slipstream reactor just downstream of the knife valve. Below the 4-inch inlet pipe, the SS pipe will change from round to rectangular to enter the catalyst housing. The housing is ~20" x 15" x 50" long. On the bottom of the catalyst housing is a bank of venturi flowmeters and eductors to draw the exhaust gases through the apparatus. From here the shell returns to 6" ID low-carbon steel pipe that returns the gases to the system downstream of the air heater.

The reactor is designed to require as little operator intervention and maintenance as possible. Operation of the slipstream reactor will be monitored by REI via remote access of the control system using a computer network. REI will notify the plant of any major changes in status or operation of the reactor. REI will arrange for delivery of ammonia tanks to the plant. Approximately every six weeks, REI personnel will be on-site to remove catalyst samples and change ammonia tanks as needed.

The plant will supply boiler operating data to REI taken during the test period on a weekly basis. The plant will take monthly samples (~1 liter each) of fuel and ash. REI will not disclose this information publicly without permission from the plant. Monthly coal and ESP hopper ash samples will also be taken.

Slipstream Reactor Control System

The testing plan calls for the reactor to be operated continuously at remote power plant sites, under harsh environmental conditions for a period of six months, with very little if any on-site human interaction. These requirements called for an autonomous, rugged, reliable and remotely accessible control system that could be implemented under the budget constraints of the project. In order to meet these goals, REI chose National Instrument's LabVIEW RT running on a FieldPoint FP-2010 module with analog, digital and temperature modules providing I/O capabilities. RT handles numerous PID control loops, error and warning checking and data transfers to a host PC. The host PC, located in the office building at the plant, runs LabVIEW 6.1 and handles web-based user interaction and data logging.

The reactor operates as follows: When the pneumatic gate valve on the inlet of the reactor is open, flue gas from the power plant flue gas duct is allowed to enter the reactor. Upon entering, the nitrogen oxide concentration in the gas stream is measured using a gas analyzer. Using this information, a mass flow controller connected to ammonia storage tanks injects a stoichiometrically appropriate amount of reagent into the stream. From here, the gas enters six catalyst chambers, each with a different type of catalyst. The flow rate through each of the chambers is controlled using eductors (which create low pressure in the chambers) and associated pressure control valves, which drive the eductors. Feedback for this control comes from six venturi flow meters connected to differential pressure transducers. This system allows the flow rate through each of the chambers to be controlled independently as required for different catalyst types and conditions. Since the flow rates through the catalysts are not independent of one another, an iterative approach was used where PID is applied to each chamber several times in sequence. This sequence through the chambers is then repeated to convergence. After leaving the chambers, the gas is returned to the flue duct.

The temperature of the reactor is closely monitored and controlled using electric heaters and thermocouple measurements taken at various locations within the reactor. Flue gas flowing through the reactor is sampled to measure oxygen, nitrogen oxide and carbon dioxide both before and after the catalysts. This data defines the effectiveness of the chemical reactions in the catalysts. A sootblowing system minimizes ash buildup and maintains catalyst activity.

Figure 18 shows a diagram of the control system. It consists of an on-site PC, a bank of NI Fieldpoint modules (including an FP-2010), differential pressure transducers, thermocouples, solenoid control valves, pressure control valves, a mass-flow controller and gas sampling equipment. The control software consists of an RT application running on the FP-2010 and LabVIEW 6.1 on the host PC.

Figure 18. Conceptual diagram of the control system

RT Application: The heart of the control software lives on the Fieldpoint RT module. This software controls all functions of the reactor including the state machine logic, temperature control (PID), ammonia control, chamber flow control (PID), data acquisition, communication and error handling. The RT software runs independently of the host PC and is not subject to the instabilities and indeterminism often associated with the Windows operating system.

A state machine provides the intelligence for the RT software. It provides a high degree of autonomous control, with the ability to recover from a range of error conditions and, if necessary, to shutdown the reactor and inform personnel of the problem. This error handling is important since we use ammonia, which is dangerous if released.

Host PC Application: This application logs data broadcast from RT and provides both a local and remote user interface for the control software. Transfer of data from RT to the host occurs via a datsocket connection. These data blocks are sent at a user specified interval and contain information regarding reactor state, RT software status etc. User interface controls on the RT virtual instruments made available to users (items such as PID parameters, state machine status etc) are set using VI server calls. Remote interaction with the host occurs using a web browser. This was trivial to implement using LabVIEW 6.1's web publishing capabilities.

When an unrecoverable error is read from RT (such as a component failure), the host application sets indicators and sends out email. Although the host provides information about the critical error to the user, RT actually handles the error.

Figures 19 and 20 show views from the host PC application VI. The Status view shows data coming from RT such as temperatures, pressures, errors and warnings and state machine status. Elements on this view are read-only. The Control view allows the user to make changes to RT such as the state of the state machine, clear error, setpoints, and sootblowing and ammonia parameters. Views not shown in these figures are used for error and warning ranges, host configuration and manual operation, etc. More detail on the control system is provided in the Appendix to this report.

Figure 19. Host PC LabVIEW application **Status**

Figure 20. Host PC LabVIEW application **Control** view

Slipstream Reactor Operation

The site of the first slipstream reactor test, AEP's Rockport plant, consists of two 1300 MW_e B&W opposed wall-fired boilers. These are supercritical boilers that burn a blend of bituminous and subbituminous coals. During this quarter the slipstream reactor was successfully installed at the Rockport plant on Unit 1, Air Heater #2. Figures 21 through 23 show pictures of the reactor during and after installation. Personnel from REI and the University of Utah worked with plant personnel to install the reactor. REI personnel completed connection of the reactor control system to a local host computer; this host computer was connected to the Internet through the plant's Local Area Network. REI verified that the SCR reactor can be accessed remotely from REI's offices in Salt Lake City; using remote access, operating data can be downloaded, settings can be changed and the control system program can be updated. An operating manual was prepared for the plant containing procedures and detailed information on the control system; this can be found as an Appendix to this report.

One of the outstanding issue remaining is safe handling of ammonia at the plant. Ammonia will be delivered to the plant in 150-lb cylinders. This is liquid, anhydrous ammonia in the cylinders; once the pressure is released, it is converted to gaseous ammonia for delivery to the reactor. We will need approximately 50 lb of ammonia per week. Thus, the 600 lb should last about 12

weeks. The ammonia manifold that is part of the slipstream reactor system is set up to handle 4 cylinders, with two at a time connected to the system. At the estimated usage rate, therefore, the valves on the manifold would need to be switched every six weeks. At twelve weeks, the cylinders would need to be changed. Approximately every six weeks, REI personnel will be on-site to remove catalyst samples and change ammonia tanks as needed. We do not plan to have more than 600 lb of ammonia on site at any time. As catalyst sample is withdrawn and the flow through the reactors decreases and so does the ammonia requirement.

Figure 21. Installation of the SCR flue gas intake pipe at Rockport.

Figure 22. SCR Unit installed between the economizer and the air heater at Rockport Plant.

Figure 23. CEMS and SEQUENCER cabinets installed at Rockport Plant.

Results and Discussion

Soot: During this quarter, progress was made on the computational simulation of a full-scale boiler with the purpose of understanding the potential impacts of burner operating conditions on soot and NO_x generation. Burner stoichiometry can have a significant impact on soot formation/destruction as well as on NO_x emission. The results of full-scale application illustrate that the limited mixing in the system can cause locally oxygen deficient zones, resulting in inefficient destruction of soot even at high temperature (> 1500 K) and potential soot emission in flue gas. The level of detail provided by the simulations can be a valuable aid in understanding the mechanisms by which combustion modification affects soot formation/destruction and NO_x emission.

SCR Catalyst: During this quarter, in situ analyses of vanadium-based SCR catalyst systems were completed at BYU. These are preliminary data primarily intended as shakedown for the system. Results to date indicate that the system produces results that represent improvements compared to literature examples of similar experiments (but for different catalyst systems). Investigations of surface chemistry specific to vanadium-based SCR catalysts show mixed results. We have as yet been unable to reproduce surface sulfation described by some authors in the literature. However, we have reproduced substantial ammonia and NO adsorption data that are consistent with literature sources. Construction of the CCS reactor system is essentially complete and the control hardware and software are largely in place. A problem encountered with ammonia analyzer interferences is close to being resolved. A large batch of vanadia/titania catalyst in powder form has been prepared for using in poisoning tests. An experimental design for poisoning of this catalyst with calcium and sodium minerals over a range of concentrations has been formulated.

During this quarter, minor modifications were made to the reactor and to the control system. The slipstream reactor was installed at AEP's Rockport plant at the end of November 2002. An operating manual for the reactor was written and given to the plant. The outstanding issues that need to be resolved concerning the Rockport testing are as follows:

- What needs to be done to start the reactor (without ammonia):
 - Host Site Agreement must be approved and signed by AEP.
- What must be done to hook up the ammonia:
 - Rockport must write up emergency response procedures for ammonia, carry out general worker awareness, and conduct more detailed training for the individuals who will be directly involved with the reactor (they can handle this).
 - Rockport will run vent lines from the ammonia manifold to outside the building.
 - REI must install shut-off valves on individual ammonia cylinders and labels for ammonia lines and valves on the manifold.

When the Host Site Agreement is executed, then REI will begin long-term shakedown of the reactor; this can take place without ammonia. In parallel, REI is negotiating with DOE the contract for the mercury oxidation tests to be carried out on the slipstream reactor.

Conclusions

Good progress has been made on several fronts during the last three months. In particular:

- The results of full-scale application of the soot-formation model integrated into a CFD code illustrate that the limited mixing in the system can cause locally oxygen deficient zones, resulting in inefficient destruction of soot even at high temperature (> 1500 K) and potential soot emission in flue gas. The impact of burner stoichiometry can be significant on soot formation/destruction as well as on NO_x emission. The level of detail provided by the CFD simulations can be a valuable aid in understanding the mechanisms by which combustion modification affects soot formation/destruction and NO_x emission.
- During this quarter, *in situ* analyses of vanadium-based SCR catalyst systems were completed. These are preliminary data primarily intended as shakedown for the system. Results to date indicate that the system produces results that represent improvements compared to literature examples of similar experiments (but for different catalyst systems). Investigations of surface chemistry specific to vanadium-based SCR catalysts show mixed results. We have as yet been unable to reproduce surface sulfation described by some authors in the literature. However, we have reproduced substantial ammonia and NO adsorption data that are consistent with literature sources.
- Construction of the CCS reactor system is essentially complete and the control hardware and software are largely in place. A problem encountered with ammonia analyzer interferences is close to being resolved.
- A large batch of vanadia/titania catalyst in powder form has been prepared for use in poisoning tests. An experimental design for poisoning of this catalyst with calcium and sodium minerals over a range of concentrations has been formulated.
- Minor modifications were made to the slipstream reactor and to the control system. The slipstream reactor was installed at AEP's Rockport plant at the end of November 2002. Details of ammonia handling and safety training remain to be completed; REI is working with the plant to complete these. AEP must also review and sign a Host Site Agreement before the testing can begin. In parallel, REI is negotiating with DOE the contract for the mercury oxidation tests to be carried out on the slipstream reactor.

Plans for the next quarter include: beginning of catalyst poisoning experiments at BYU; continuation of *in situ* studies of surface compounds on SCR catalyst; and start-up of the slipstream reactor at Rockport plant.

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APPENDIX:
SLIPSTREAM REACTOR OPERATING PROCEDURES

TABLE OF CONTENTS

Section	Page
1 INTRODUCTION	2
2 OBJECTIVES	2
3 REACTOR SYSTEM DESCRIPTION	3
<i>a. Overview</i>	3
<i>b. Flow System</i>	4
<i>c. Catalyst Arrangement</i>	6
<i>d. Sampling and Control</i>	7
<i>e. Location at the Boiler</i>	9
4 STARTUP AND SHUTDOWN PROCEDURES	12
<i>Startup</i>	12
<i>Shutdown</i>	14
<i>Startup and Shutdown Procedures During an Outage</i>	15
5 APPENDICES	16
A. SERVICES REQUIRED	16
<i>Plant Air</i>	16
<i>Cooling Water</i>	16
<i>Computer and Telephone Lines</i>	16
<i>Power</i>	16
B. SAFETY	17
<i>a. Safety Systems</i>	17
<i>Reactor Shutdown</i>	17
<i>Chemical Safety</i>	17
<i>b. Ammonia Safety</i>	17
<i>Ammonia Leaks</i>	17
<i>Ammonia Safety Awareness</i>	18
<i>c. Personnel Safety</i>	19
<i>REI Personnel</i>	19
<i>Plant Personnel</i>	19
C. INSTALLATION DECOMMISSIONING AND REMOVAL	20
<i>a. Installation</i>	20
<i>b. Decommissioning and removal</i>	20
D. SOFTWARE DESCRIPTION	20
<i>a. SCR Controller Configuration</i>	20
<i>b. Software Architecture</i>	20
<i>c. Software Display Panels</i>	24
<i>d. Summary of Data Logged</i>	37
E. SCR EQUIPMENT OPERATION AND INSPECTIONS	39
<i>a. Suggested List of Equipment Checks</i>	39
<i>b. Ammonia Usage and System Maintenance</i>	40
<i>c. Equipment Log sheets</i>	43
<i>d. Notes on Equipment Checklist</i>	47
F. CONTACT INFORMATION	48
G. ANHYDROUS AMMONIA MATERIAL SAFETY DATA SHEET	49

1. INTRODUCTION

Reaction Engineering International (REI) has been funded by the US Department of Energy (under cooperative agreement DE-FC26-00NT40753) to carry out a program titled “NO_x Control Options and Integration for US Coal Fired Boilers.” The overall goal of the program is to evaluate and demonstrate low cost NO_x control strategies and to study the potential impact of these NO_x control technologies on overall electric utility boiler performance. REI’s investigations have included combustion modifications and downstream clean up.

The cost and effectiveness of NO_x control strategies for coal-fired boilers has received considerable attention over the last two decades and a number of successful approaches have been applied. Utilities must weigh a number of issues when determining the best strategy for their specific units. Comparisons routinely include cost and the amount of NO_x reduction, with selective catalytic reduction (SCR) often used as a standard for comparison. However, in order to make this comparison meaningful, the total cost of combustion modifications must be considered (including such things as carbon-in-ash levels, waterwall wastage, etc.). Industry is developing experience evaluating many of these costs and it is one of the tasks of this program to better understand two such areas (waterwall corrosion and soot formation). For SCR systems, the “real” cost for coal-fired boilers using US coals and coal/biomass blends is very uncertain.

2. OBJECTIVES OF THE FIELD TEST PROGRAM

One of the tasks in REI’s program is called “SCR Performance Under Co-firing Conditions.” The overall objective is to evaluate SCR costs at a deeper level and to improve estimates of actual costs. One of the prime motivations for this program is to look at the influence of the alkali and alkaline earth elements in biomass and how those affect SCR catalyst during firing of Powder River Basin (PRB) coal or when biomass is co-fired with coal.

The end result of the program will be the development of a public-domain sub model that describes SCR deactivation by low-rank coals and biomass. This sub model will be based on existing data and the data generated under this project and will be sensitive to alkali and alkaline earth content, gas-phase moisture content, catalyst type and physical construction, ammonia concentration and operating temperature among other things. The model will predict NO_x conversion, ammonia slip and SO₂ conversion.

Measured data on the activity of catalyst subjected to varied environments are critical to development of the deactivation model. The goal of the field-testing task is to test up to four commercial vanadium-based catalysts in a flow reactor in which flue gas from a coal-fired power plant circulates through the reactor at the appropriate temperature and into which ammonia is injected. The flow reactor has been built at the University of Utah. Flow reactor testing will be carried out at the plant for approximately six months. Every 6 weeks, a sample of each catalyst will be removed and sent to Brigham Young

University (BYU) for analysis of activity. NO_x removal will also be measured on-line across each catalyst using a dedicated CEM system.

3. REACTOR SYSTEM DESCRIPTION

The SCR reactor set up consists of the slip-stream reactor referred to as the SCR unit, a data acquisition and control enclosure (CONTROL BOX), an air supply controls enclosure (AIR BOX), an enclosure for sample conditioning and switching between lines (SEQUENCER) and a cabinet for housing the NO_x and O₂ continuous analyzers (CEMS). In addition the reactor is connected to an ammonia supply manifold and a suite of calibration gases. The complete system for the slipstream reactor consists of the equipment listed in Table 1. The following is a brief description of these components.

a. Overview

The slipstream reactor must meet the following requirements:

- Withdraw flue gas from the boiler at a point between the economizer exit and the air preheater inlet with a minimum temperature of 350 °C (660 °F);
- Provide a reasonably accurate representation of the fly ash at the inlet to the slipstream reactor, particularly of the sub micron fly ash (which has been implicated in masking of catalyst exposed to PRB ash);
- Inject anhydrous ammonia at the inlet to the catalysts based on the inlet NO_x concentration;
- On-line measurement of NO_x, at the inlet to the reactor and at the outlet of each individual catalyst chamber;
- Maintain constant gas velocity across the catalyst (i.e., constant volumetric flow rate) for the duration of the test;
- Remove ash accumulation at the leading edge of the catalyst periodically to maintain the proper flow through the catalyst;
- Maintain the catalyst above the acid gas dew point to prevent condensation on the catalyst;
- Provide the ability to remove parts of the catalyst to be sent back to BYU for characterization;
- Return the flue gas back into the duct once it leaves the slipstream reactor;
- Operate safely in the power plant environment with minimum maintenance and high availability for approximately six months at a time;
- Communicate remotely with REI for the purposes of monitoring operation, logging data and changing control settings.

Table 1. Components of SCR Slipstream Reactor

Quantity	Item
1	SCR Reactor
1	CONTROL BOX
1	AIR BOX
1	CEMs
1	SEQUENCER
4	Ammonia cylinders
3	Calibration gas cylinders
1	Local PC (located in office space)

b. Flow System

Figure 1 shows a schematic drawing of the reactor. There are six identical chambers. The overall flow through the system is controlled by a single eductor just upstream of the system exhaust. A venturi flowmeter at the inlet to the system provides feedback to a controller that adjusts a valve on the air line to the eductor. The eductor has a large flow range and turndown ratio. To ensure that the flow rates through the catalyst chambers are equal, the pressure drops are set equal by adjusting the flow of compressed air through eductors downstream of the catalysts. Air flow to the eductors flows through a set of six differential pressure control valves located in the AIR BOX.

Anhydrous ammonia will be injected into the flue gas stream, and blended with a static mixer, housed in a spool piece located above the catalyst chambers. The ammonia feed rate will be maintained by a mass flow controller, and will be adjusted based on the measured amount of NO_x entering the system. The molar ratio of NH_3/NO_x will be set at approximately 1.1 in order to ensure that NO_x destruction is not limited by stoichiometry and to give margin for swings in NO_x levels.

Ammonia will be delivered to the plant in 150-lb cylinders. This is liquid, anhydrous ammonia in the cylinders; once the pressure is released, it is converted to gaseous ammonia for delivery to the reactor. We will need approximately 50 lb of ammonia per week. (This may change once we see what the plant NO_x levels actually are). Thus, the 600 lb should last about 12 weeks. The ammonia manifold to be supplied by REI is set up to handle 4 cylinders, with two at a time connected to the system. At the estimated usage rate, therefore, the valves on the manifold would need to be switched every six weeks. At twelve weeks, the cylinders would need to be changed. Approximately every six weeks, REI personnel will be on-site to remove catalyst samples and change ammonia tanks as needed. We do not plan to have more than 600 lb of ammonia on site at any time. As catalyst sample is withdrawn and the flow through the reactors decreases and so does the ammonia requirement.

Figure 1. Schematic of SCR Slipstream Reactor

c. Catalyst Arrangement

Six catalysts will be tested; four monolith and two plate, and will be configured as shown in Figure 2.

Figure 2. Arrangement of catalysts (plan view)

The four monolith catalysts will be installed in four sections each. Each section will have a cross section of 2.25 by 2.25 inches, and will be housed in a 48-inch long aluminum square tube with outer dimensions of 2.5 by 2.5 inches and 1/8" wall thickness. The overall cross section of each monolith catalyst will be 4.5 x 4.5 inches. The four tubes will be bunched together as a square with outer dimensions 5.0 x 5.0 inches.

The plate catalysts will be housed in square aluminum tubes with an inside dimension of 4.75 inches (5.0 inches outside, 1/8" wall thickness). Roughly 20 plates will be placed in the tube, resting in slots along opposite walls. This configuration will have the same outer dimensions as the groupings of monolith catalysts.

Catalyst samples will be taken three times during the testing period in order to diagnose deactivation. The total flow through the system will be adjusted downwards as catalyst samples are removed. The total cross sectional area of the two plate catalysts will remain the same throughout the test. Flow channels of the four monolith catalysts will be blocked off as catalyst samples are taken. The overall flow rate will be adjusted to correspond to the cross sectional area of the remaining catalyst chambers, and will follow the progression given below:

Initial configuration:	100.0%
After first sample has been removed:	83.3%
After second sample has been removed:	66.7%
After third sample has been removed:	50.0%

Samples of monolith catalyst will be taken by removing one of the four chambers entirely. The opening that remains will be blocked off at the inlet by a sloped cover to minimize ash deposition and flow disturbances.

Figure 3. Catalyst removal plan.

Plate catalyst samples will be taken by removing every fourth plate, and replacing it with a non-reactive dummy plate.

The order of catalyst removal is shown in Figure 3. Blocked monolith chambers are shown by a gray square. Non-reactive dummy plates are shown in gray. Monolith chambers are removed starting from the outside in order to keep the profile as centered and uniform as possible.

The open cross section of the catalyst system decreases as samples are removed. To keep the flow velocity constant through the chambers, the overall flow through the system is decreased correspondingly as catalysts are removed, as described above in the section on the flow control system. In terms of the initial flow through the system, the overall flow follows the progression 100%, 83%, 67%, 50%. The pressure drops across the six catalyst sections (divided by catalyst type) are adjusted with butterfly valves so that they are identical to each other (as described above), so the flow

velocity per catalyst chamber should be the same throughout the duration of the tests.

d. Sampling and Control

In order to minimize ash buildup in the system and maintain catalyst activity, large volumes of high-pressure air are periodically blown through the system. A line connected to the plant's compressed air system (~100 psig) runs to an automatic open/shut valve. This supplies air to a bank of "soot-blowing" nozzles placed above the catalyst chambers. When open, air is blown downwards into the catalyst chambers for about 10 seconds. The volumetric flow rate of this air is several times higher than that normally flowing through the system, and will blow out any ash that may have deposited. Prior to activating the "soot-blowing" sequence, the sampling system is isolated.

The system has seven sampling ports, one before the catalyst chambers and one after each of the six catalyst sections. The ports themselves consist of thin tubes that enter the channel and bend downwards, in line with gas flow. Each sampling port is fitted with a stainless steel frit for removal of particles laden in the sample. A sample drawn either from the SCR inlet or exit via a heated stainless steel line is sent to a sample-conditioning module housed in an enclosure called the SEQUENCER. The sequencer houses a sampling pump, a thermoelectric cooler for removal of moisture, candle filter and a bank of solenoid valves for sample line selection. Through an automatic valve-switching sequence set in the software, one of these gases at a time is sent to the CEM via heated stainless steel line. In the sequencer, provisions are also made of blowback of the sample lines. The CEMs consist of a Horiba Model CLA-510 Chemiluminescent NO-NO_x Analyzer and a Horiba Model MPA-510 Magnetopneumatic Paramagnetic O₂ Analyzer. In order to maximize accuracy, the pre-catalyst gas is analyzed before each post-catalyst measurement. Thus, analyses are performed in the order: pre, post 1, pre, post 2, pre, post 3, etc. The data from the analyses are logged and can be remotely accessed via modem or network.

The control system, located in a NEMA 4 enclosure (CONTROL BOX), receives inputs from the reactor, sends outputs to the reactor for control signals and communicates with a PC, as shown below.

Figure 4. Control System

The dashed line drawn around the reactor indicates the control system (consisting of the CONTROL BOX and AIR BOX) that holds the electronic components, including the microcontroller and associated I/O, relays, power supplies, and power conditioning. This box will be physically located next to the reactor and will have to be protected from the elements. The PC will be located in the control room or office area as designated by the plant. In addition, REI can access the control system remotely. The control hardware is based around a National Instruments Fieldpoint system.

e. Location at the Boiler

The reactor flue gas intake will be attached to boiler at the economizer outlet where up to 250 scfm of gases will be withdrawn through a probe (Figure 5) inserted in an existing port. The probe is also fitted with a thermocouple for monitoring the temperature of the flue gas as it exits the economizer. After passing through the reactor, the gases will be exhausted through an existing port at the air heater exit. The reactor itself is approximately 8 feet long, with a 25 x 30 inch footprint, and weighs approximately 1000 lbs. The reactor will be well insulated and securely fastened.

Anhydrous ammonia (stored in cylinders that hold approximately 150 lb each) is introduced into the slipstream reactor just downstream of the knife valve. Below the 4-inch inlet pipe, the SS pipe will change from round to rectangular to enter the catalyst housing. The housing is ~20" x 15" x 50" long. On the bottom of the catalyst housing is a bank of venturi flowmeters and eductors to draw the exhaust gases through the apparatus. From here the shell returns to 6" ID low-carbon steel pipe that returns the gases to the system downstream of the air heater.

Four ammonia cylinders, connected together and to the reactor system using a manifold, will be located at ground level. At the moment these are located in the Fan room. Three tanks of calibration gas for the CEMs will be located near the SEQUENCER and the CEMs.

Figure 6 is a photograph of the slipstream reactor, undergoing shakedown testing at the University of Utah. In this picture, the doors covering the catalyst chambers were removed and the reactor was not insulated, as would be the case at the plant.

Figure 5. Boiler suction tube assembly.

Figure 6. Slipstream reactor (without doors on catalyst chambers and insulation).

4. STARTUP AND SHUTDOWN PROCEDURES

Start up

The procedure to start the reactor is as follows:

At the Reactor.

- (1) Make sure that power is supplied to the CONTROL BOX, the AIR BOX, the CEMS and SEQUENCER
- (2) Power up the heated sample lines from red switches inside the back of the SEQUENCER. All the sample line switches should light up.
- (3) Open the plant air supply valve.
- (4) Open the manual air supply valve which supplies air to the pneumatic gate valve.
- (5) Inspect the two inlet and outlet three-inch blind flanges located on the SCR intake and exit lines. These should be shut.
- (6) Open the manual gate valve for the SCR exit gas flow into the flue gas duct.

At the Ammonia Station.

- (1) Check whether the Ammonia cylinder pressure is less than **125 psig**.
- (2) If the ammonia cylinder pressure limit is exceeded, open the manual vent valves and other appropriate valves in the cylinder line to release ammonia until the desired pressure level is reached.
- (3) Make sure that the ammonia manifold vent valves are shut.
- (4) Now open the ammonia cylinder valve.
- (5) Open the manifold valves to direct flow to the SCR reactor making sure that ammonia pressure in the transfer line does not exceed **85 psi**.

At the PC.

- (1) Start the SCR control software
- (2) Set the SCR reactor panel temperature (650°F)
- (3) Make sure the correct minimum and maximum catalyst chamber temperatures have been set.

- (4) Set the appropriate flow rate in each catalyst chamber. (Total flow rate is 250 cfm initially; flow rate will decrease as catalyst is removed.)
- (5) Select the desired state (idle, run, inactive, etc.).
- (6) Push the “set state” button to initiate execution of the selected state.

Details of the software for operating the reactor can be found in the Appendix. It may be noted that ***plant personnel may be asked to perform the tasks listed above at the reactor, at the ammonia station and at the PC, should the need arise.***

The reactor is designed to require as little operator intervention and maintenance as possible. Operation of the slipstream reactor will be monitored by REI via remote access of the control system using a computer network. REI will notify the plant of any major changes in status or operation of the reactor. REI will arrange for delivery of ammonia tanks to the plant. Approximately every six weeks, REI personnel will be on-site to remove catalyst samples and change ammonia tanks as needed.

The ammonia tanks should be visually inspected by plant personnel at least on a weekly basis. The gas cylinders for calibration of the CEM should be visually checked by plant personnel on a weekly basis. Filters and flow meters in the CEM system should be visually inspected by plant personnel several times per week. Automatic scheduled blowback of all sample points should keep the CEM sample lines free of particulate matter. Filters will be changed, if needed, by the plant and replaced by new filters supplied by REI. The sample pumps contain diaphragms that are normally replaced on a quarterly basis; this will be done by REI.

The plant will supply boiler operating data to REI taken during the test period on a weekly basis. The plant will take monthly samples (~1 liter each) of fuel and ash. REI will not disclose this information publicly without permission from the plant. These data include readings of the following, if available:

- Unit load, MW (gross and net)
- Total Air Flow
- Primary Air Flow (Air flows may come from fan curves and damper positions)
- Secondary Air Flow
- Total Coal Feed
- Individual Burner Coal Feed (Coal feed rates may be from gravimetric feeders)
- Individual Burner Air Flow
- Biomass Feed Rate
- Furnace Exit gas Temperature
- Economizer Exit Temperature
- Pressure Drop Across Air Preheater
- Boiler Exit Oxygen Levels
- Stack Flow Rate
- Opacity
- All available SEM Data (NO_x, CO, O₂, CO₂)

REI will remove catalyst samples three times (roughly every six weeks) during the testing period in order to diagnose deactivation. For this operation, REI will have one person on-site to remove the catalyst.

Shutdown

An *automatic emergency* shutdown will be triggered by any of the following events singly:

- Loss of AC power to the system
- Loss of 24 VDC power from any of the power supplies
- Low temperature at the duct entrance (Less than 600°F)
- Low flow in the reactor
- Failure of the reactor heaters
- Loss of ammonia, as detected by the mass flow controller or loss of ammonia pressure, as detected by the ammonia supply pressure
- On command through the control system's user interface, either locally or remotely

Since the system will have an uninterruptible power supply (UPS), the shutdown procedure will change the system from Run mode to Inactive mode using the following sequence:

- Issue a warning to the user interface that a shutdown has been initiated
- Stop sootblowing (if in progress)
- Stop sampling (if in progress)
- Close the ammonia shut-off valve
- Deactivate the ammonia flow control loop
- If a loss of power was not detected, ramp the heaters down to the Idle set point.
- The system is now in Idle mode.
- From Idle mode, deactivate the chamber flow control loops and turn off the eductor control valves
- Open the purge air valve
- Close the gate valve
- If there is power to the heaters, ramp the temperature down
- Deactivate the heater control loops.

Alarms. Alarms will be displayed on the user interface (local PC) and logged with data. In one case, an alarm will initiate action: if the inlet temperature to the catalyst chamber falls outside the set range for example a minimum of 600 °F and a maximum of 750°F. If any of these set temperature limits is violated, then the ammonia will be turned off until the desired temperature range is restored. When an error occurs, the system will send an e-mail automatically to designated personnel at REI. Table 2 shows some suggested error ranges for flue gas entrance temperature, cabinet temperature, and ammonia supply pressures. An example of an error panel is shown in the appendix.

A **voluntary** shutdown of the reactor can be achieved by accessing the control panel of the software (at the PC) and selecting the “**Inactive**” state in the Target State pull-down menu. This can be done either at the resident computer located at the plant site next door to Steve Pfeister’s office or remotely from a computer in REI’s office in Salt Lake City, Utah. Alternatively, the reactor can be shutdown by switching off the mains power to the entire SCR system set-up. The SCR gets its power from a transformer located next to the CEMS. The switch can be identified by tracing the power supply cable to the transformer.

Table 2. Suggested error limits for flue gas entrance temperature, catalyst cabinet temperature, and ammonia supply pressures.

Error Level	Controlled Variable		
	Gas entrance temperature	Catalyst cabinet temperature	Ammonia supply pressure
Low	600°F	600°F	20 psi
High	800°F	750°F	85 psi

Startup and Shutdown Procedures During an Outage

Shutting down for an outage. In the event of an outage caused by an emergency, the system will shut itself down as explained in the **shutdown** section above. If the outage is anticipated, then a voluntary shutdown procedure can be launched as explained above in the **voluntary** shutdown section. In either case, the following actions must follow-up:

- (1) Isolate the ammonia supply system by shutting off valves at the cylinder and manifold uptake lines. To ensure that no ammonia flows inadvertently during an outage and enter areas of outage maintenance activities; shut off all valves in the ammonia transfer lines at the manifold.
- (2) Bleed the ammonia remaining in the lines by opening the manifold vent valve until the ammonia cylinder delivery pressure gauge reading declines to zero.
- (3) Shut off the ammonia vent valves after completing the vent procedures.
- (4) The system should shut off in the manner of the checked items above.
- (5) Shut off the SCR manual **outlet** gate valve.
- (6) If the outage is going to take less than 2 days, then there is no need to isolate the panel heaters. These will continue to heat-up the SCR chamber maintaining its temperature at 600°F in an **inactive** state. There is also no need to shut off air to the solenoid-operated gate valve and power to the sample line heaters. But if the outage is expected to last more than 2 days, then shut off air to the solenoid-operated gate valve, and shut-off power to the sample line heaters.
- (7) To prevent moisture condensation on the catalyst, at the PC, set the SCR reactor panel temperature at 300°F.
- (8) If the Outage occurs in a weekday and within office hours (9am-5pm Mountain Time) contact REI personnel for assistance, else, follow the procedures outlined.

Starting up after an Outage. The startup procedure after an outage is the same as that described at the beginning of this section. Contact REI personnel before starting up.

5. APPENDICES

A. SERVICES REQUIRED

Plant Air

The eductors, which control the flow through the reactor, require a maximum of 100 scfm of plant air at about 100 psig pressure. In addition to this steady flow of air, the soot blowing system will require 250 scfm of air for short (10 s) periods of time at least once per day; the reactor intake pneumatic valve and the sample line blowback system will also periodically require plant air.

Cooling Water

No cooling water is required.

Computer and Telephone Lines

The microcontroller that controls the reactor will be located in the control box at the reactor itself. There will be an Ethernet cable connecting the microcontroller with a personal computer (PC) located in a control room or office area to be specified by the plant. The cable has a maximum length of 300 feet. If the distance between the reactor and the control room is greater, a repeater will be used, which will require a small amount of electrical power. The PC will communicate with the outside world via a secure connection to the plant's network (the preferred option) or via a phone line. REI will monitor the operation of the reactor in this manner.

Power

The CEM systems and most of the slipstream reactor require 115 or 120 VAC. The reactor also requires 240 VAC single-phase electricity to power the strip heaters mounted on the reactor enclosure panels. Since only 600 VAC is available and a 600-to-480 transformer is in place, the plant will supply a transformer to convert 480 VAC to 240 VAC. Power requirements for the reactor are given in the Table A1.

Table A1. Power Requirements

	120 VAC	240 VAC
SCR Reactor	20 A	40 A
CEM System	15 A	---
Hg Measurement	20A	

B. SAFETY

a. Safety systems

Reactor Shutdown

As discussed in “System Startup and Shutdown Procedures” section, the system will have an uninterruptible power supply (UPS) in order to handle short-term loss of power. The system will automatically shut down for longer power outages, for loss of compressed air, for loss of ammonia, or for low temperature at the flue gas inlet.

Chemical Safety

Material Safety Data Sheets (MSDS) will be provided to the plant for ammonia and for the catalysts. Note that the threshold ammonia values for reporting to federal agencies are 10,000 lb (OSHA Process Safety Management) and 20,000 lb (EPA Risk Management).

Ammonia will be delivered to the plant in 150-lb cylinders. This is liquid, anhydrous ammonia in the cylinders; once pressure is released, it is converted to gaseous ammonia for delivery to the reactor. We will need approximately 50 lb of ammonia per week. (This may change once we see what the plant NO_x levels actually are). Thus, the 600 lb should last about 12 weeks. The ammonia manifold to be supplied by REI is set up to handle 4 cylinders, with two at a time connected to the system. At the estimated usage rate, therefore, the valves on the manifold would need to be switched every six weeks. At twelve weeks, the cylinders would need to be changed. We do not plan to have more than 600 lb of ammonia on site at any time. If requested by the plant, the ammonia supply company could organize safety training for plant personnel either for a one-hour session or a 24-hour course with full hands-on training.

The maximum ammonia cylinder pressure is 480 psi. However, safety relief valves are included in the ammonia manifold which will not permit the cylinder pressure to rise above 250 psi. If this pressure is reached the valves will open to vent the ammonia.

b. Ammonia Safety

Ammonia Leaks.

An ammonia leak is easily detected by the sharp, pungent odor of the vapor. All leaks should be approached with caution. When a leak is detected, the first step should be to determine the size of the leak, its location and whether it is continuous or single release such as would occur when a hose is vented before being disconnected.

Leaks may be accurately located by using sulfur tapers or moist litmus test papers. Where small leaks are detected, they should be repaired. Leaks never get better; they always get worse. Don't wait for an emergency situation to occur before fixing leaks or requesting assistance.

Vapors from small leaks may be absorbed by covering the area with a water-soaked towel. The towel must be rinsed out and replaced when saturated. Rubber gloves must be worn. When working on ammonia vapor leaks, stand upwind from the leak. Indoors, a portable fan is useful to disburse the vapors.

Many small leaks are a result of seasonal temperature fluctuations. There are various components and material used in an ammonia system. Since many of these materials have different rates of expansion and contractions (i.e., elastomers used as gaskets and steel used a valve body) the result can be occasional leaks in areas these materials come into contact.

Small leaks can often be corrected by tightening a nut or a bolt as in the case of an ammonia flange. Valve stem leaks (where no packing nut is available) can often be corrected by the application of a penetrating oil (WD-40) and opening and closing the valve a few times. Leaks at the float gauge gasket may be eliminated by tightening the four screws. First, loosen one, apply some penetrating oil, and then tighten. Next, loosen and tighten the screw, then the remaining ones. If this does not work, the gasket must be replaced and a “pump-out” is required.

Where relatively large leaks are encountered, eliminate the ammonia source, if possible, by closing off a valve. If the situation is beyond your control call TANNER INDUSTRIES for assistance at 1-800-643-6226. In the unlikely event that they cannot be reached contact CHEMTREC, the Chemical Emergency Center at 1-800-424-9300.

Ammonia Safety Awareness

It is important that personnel understand the properties of ammonia and that they be thoroughly trained in safe practices for its storage and handling.

Please refer to the MATERIAL SAFETY DATA SHEET (MSDS) in the Appendix for some basic safety information. Acronyms used for Ammonia exposure limits are PEL (Permissible Exposure Limit), STEL (Short Term Exposure Limit), TLV (Threshold Limit Value), IDLH (Immediately Dangerous to Life and Health), TWA (Time Weighted Average).

The Code of Federal Regulations (29CFR1910.111) requires that all stationary storage installations have at least two suitable gas masks in a readily accessible location. Suitable gas masks are available from TANNER INDUSTRIES; INC. Gas masks must be approved by NIOSH/MSHA for use with anhydrous ammonia. Properly trained personnel can use the full-face gas mask in ammonia concentrations less than 300ppm (IDLH). Concentrations above the IDLH limit would require the use of a NIOSH approved positive-pressure, self-contained breathing apparatus (SCBA).

The Code further requires that all stationary storage installations have an easily accessible emergency shower and an eyewash unit or a 50-gallon open container with clean water.

29CFR1910.132, .133 and .134 should be consulted regarding eye and face protection and respiratory protection where employees are working directly with anhydrous ammonia.

NOTICE

The information on ammonia leaks and awareness has been compiled from TANNER INDUSTRIES' manual on "STORAGE & HANDLING OF ANHYDROUS AMMONIA" revision: 10/00.

c. Personnel safety

REI personnel

REI requires all of its employees, consultants or subcontractors who will be on-site for reactor testing to wear protective clothing, including: hard hat, safety glasses with side-shield, steel-toed shoes, and ear protection. In this special application, will wear rubber gloves, a respirator and carry at least an 8 ounce water squirt bottle while changing ammonia cylinders. In addition, REI employees, consultants or subcontractors will obey all the safety procedures required at the plant, including undergoing safety training, if requested by the plant.

Plant personnel

REI will familiarize plant personnel with the operation of the reactor before installation and will provide the appropriate plant personnel with written schematics and operating procedures for the test reactor before installation of the equipment.

C. INSTALLATION DECOMMISSIONING AND REMOVAL

a. Installation

The reactor will be installed at the exit of the economizer to guarantee an inlet gas temperature of 650-700 °F (340-370 °C). An existing four-inch port will be used to withdraw sample. A probe will be inserted into the duct, through the inlet port. The gas exiting the reactor will be injected back into the duct through a single six-inch duct downstream of the air heater. Support of the reactor will be discussed with plant personnel.

Before installation of the reactor, the plant personnel should arrange for appropriate ports in the ductwork: a four-inch port upstream of the air heater and a six-inch port downstream of the air heater in locations to be agreed upon by REI and the plant. The plant will also provide a 600 V to 240 V transformer.

REI personnel will be responsible for installation and shakedown of the slipstream reactor and CEM system. Several days of shakedown testing will be conducted to ensure the reactor is leak-tight and operating in a safe and stable fashion.

b. Decommissioning and removal

At the end of the test program, the reactor will be disconnected from the duct. Prior to decommissioning the ammonia lines will be purged to ensure safety. REI personnel will be responsible for disassembling the reactor and packing it for shipping. All structures at the plant will be returned to their original condition.

D. SOFTWARE DESCRIPTION

a. SCR Controller Configuration

The SCR control configuration consists of a FieldPoint embedded controller, a host PC located at the plant site and a remote PC located at REI's Office. A schematic of the set up is shown in Figure D1.

b. Software Architecture

The software architecture is built around parallel and non-parallel vi software modules. The parallel modules are programs that monitor data, reactor temperature, flow control, and ammonia control. The non-parallel programs include the state machine and the soot-blowing program. These are presented schematically in Figure D2. Table D1 is a summary of the LabView Vi modules and their functions.

Figure D1. Schematic representation of the SCR control.

Figure D2. Software architecture showing the entire program vi modules.

Table D1. LabView vi modules and their functions.

LabView Module	Function
Data Monitor Software	Issues warnings, error broadcasts, and NH ₃ tank pressures.
Reactor Temperature Controller	Has stable operation check to determine whether the temperature is drifting from the set point.
Flow Controller	Maintains the set point flow in each catalyst chamber as specified by the user (cfm).
Ammonia Control	Controls the flow of NH ₃ with a mass flow controller. Ammonia is dispensed based on a NO _x /NH ₃ stoichiometric ratio of 1.1.
Soot Blower	Soot blowing module. Runs in parallel but is inactive until a soot-blowing event is called.
Sample	Controls a set of relays that direct sample gas to the analyzers. Active only in RUN and RUN-NH ₃ .
Maintain	Calibrates the gas analyzers once per day. Controls the zero and span gases to the analyzer during calibration.
State Machine	Defines the system state. It cycles in 200 msec.
About Parallel vi Modules	All parallel vi's run continuously and are "dormant" until called upon by main_sm.

The state machine indicates what state the system is at any moment. There are 15 states in the control system. The current state is logged. The state machine cycles every 200 milliseconds. That is, it checks all the states for any updated instructions every 200 milliseconds. Figure D3 depicts the various states available. Soot blowing for example is the Idle Flue state. The soot blower can be triggered in 3 different ways: that is either by dp or by time of day or the user can also initiate it.

Figure D3. Schematic of states available in the main state machine (main_sm).

c. Software Display Panels

The software consists of the following display panels:

- (1) Main
- (2) Control 1
- (3) Control 2
- (4) Manual 1
- (5) Manual 2
- (6) Stability
- (7) Error
- (8) Warning
- (9) Data_log
- (10) Host

In the following each of these operational windows are described, followed by a snapshot of the relevant window.

MAIN

1. Tab control. Change to different tab menus by clicking on the desired tab.
2. Total flow rate. Displays the total flow rate through the reactor in cfm.
3. NH₃ flow rate. Displays the flow rate from the NH₃ mass flow controller.
4. The actual measured flow of ammonia into the reactor.
5. Each chamber's flow rate is displayed here in order. (1,2,3,4,5,6) It also includes a digital readout in cfm.
6. Temperature display of each chamber after the catalyst. (1,2,3,4,5,6).
7. Temperatures inside of the catalyst cabinet. 1,1R = wide side, 2,2R = wide side. North single, South single = short sides.
8. Temperature of the inlet gas when it enters the system.
9. Temperature of flue gas before it reaches the catalysts.
10. Temperature readout of the gas before it is returned to the boiler.
11. Not used.
12. Warning and error messages are displayed on this panel.
13. Current state of the system. Can be: (inactive, heat, gate_valve, purge_valve, idle_flue, init_maintenance, maintenance, init_sootblow, sootblow, run, run_nh3)
14. Critical error indicator if a fatal error is encountered. Controller is returned to the "inactive" state.
15. The state that the system is trying to reach.
16. Indicators to tell whether or not the valve is powered. When the valve doesn't have power, the gate is closed, soot is closed, purge is open, and NH₃ is closed.
17. Power indicators for incoming power. 120V 30W supply, 120V 90W supply, 120V main, 240V main.
18. Displays the current DP over each of the catalysts.

19. The control valves display what they are currently set to here.
20. NH₃ supply pressure (PSI)
21. Not used.
22. Current measurement of NO_x (PPM).
23. The sample line that is currently being analyzed. (one, two, three, inlet, etc.).
24. Air pressure difference between the compressed air line filter.
25. The current output to the heater relay. (0.0040mA to 0.0200mA).
26. Current measurement of O₂ (percent).

Figure D4. Main control panel.

CONTROL_01

1. Send the "set state" command to the FieldPoint module.
2. Clear out any critical error messages. The indicator will flash back on if the error has not been corrected.
3. Select which state you want the application to be in.
4. Control for the flow rate of each chamber. All values are in cfm.
5. Set what temperature to maintain inside the cabinet.
6. This indicator is lit up when a sootblowing event has been scheduled. This can be the result of a timed sootblow, critical pressure drop, or a manual event.
7. This button manually STARTS sootblowing.
8. This button manually STOPS sootblowing.
9. Check this box if you want the system to sootblow once per day at the given hour.
10. Check this box if you want a sootblow event to be triggered by a pressure difference.
11. The hour of the day you wish the timed sootblow to occur. This is only effective when (9) is checked.
12. Pressure at which a sootblow is triggered. This only happens if the (10) is checked.
13. This value represents the minimum amount of time between sootblowings.
14. Pressing this button updates the RT application with the new gas sampling times. Whatever values are currently in (15) (16) (17) (18).
15. Time in seconds for the span gas to flow through the analyzers during calibration.
16. Time in seconds for the zero gas to flow through the analyzers during calibration.
17. Time in seconds to wait for the current sample line to travel from the reactor to the analyzer units.
18. Time in seconds to take NO_x and O₂ readings from each chamber.

Figure D5. Control_01 Panel.

CONTROL_02

1. This is the stoichiometric ratio used in calculating how much NH_3 to inject into the inlet stream. The amount of NH_3 is determined by the following formula: *LOOK UP LATER*.
2. Enables/disables automatic maintenance. Maintenance consists of calibrating each of the analyzers once per day. NO_x and O_2 readings are taken before and after calibration for later comparison.
3. The hour of day the system will calibrate the gas analyzers. (0-24 hour).
4. This button terminates the LabView RT application running on the FieldPoint module.
5. DO NOT PRESS THIS BUTTON UNLESS ABSOLUTELY NECESSARY. If the RT application is shutdown, then the fieldpoint module must be power cycled at location in order to restart the system.
6. Not used.

Figure D6. Control_02 panel.

MANUAL_01

1. This button enables/disables manual mode. Note that when the system is taken out of manual mode, all signals from the RT application are initialized to default.
2. Turns flow control on/off. Flow control will use the values from the table on the "Control" tab. See "control_01.jpg" (4).
3. Manually write a 4-20mA signal to the control valves. Remember that the range is from 0-70cfm. (4mA = 0cfm, 20mA = 70cfm) These controls do not have any effect if flow control (2) is ON.
4. Enables ammonia control. Only enable this when the reactor is running or unless you really know what you are doing.
5. Manually open the ammonia mass flow controller. The range of the MFC is 0mL/min at 4mA to 50mL/min at 20mA. Again, don't mess with this unless you really know what you're doing!
6. Enables the reactor's temperature control. The set temperature can be found on the control tab. See "control_01.jpg" (5)
7. Manually send a 4-20 signal to the cabinet heaters. This has no effect when the reactor's temperature control (6) is activated.
8. Set of buttons to power the valves as labeled. The isolation, soot, and NH₃ valves are closed when unpowered. The purge valve is open when unpowered.
9. Toggle gas-sampling on/off.

Figure D7. Manual_01 Panel.

MANUAL_02

1. Manual control to the 4 digital signals that run the small black decoder box in the bottom of the control box. These signals control the relay bank that is connected to the sequencer. The values to the decoder are in binary. See the legend in (6) to determine which line will be opened. Note that only one line can be opened at once.
2. Direct calibration control to the gas analyzers. Both Span and Zero buttons shouldn't be pressed at the same time because only one calibration gas can flow to the analyzers at one time.
3. Activates blowback from the sequencer for all seven-sample lines.
4. Signal to the small black switch box in the sequencer. Not used.
5. Signal to the small black switch box in the sequencer. This is asserted to change the sequencer from sample mode to "cal direct" mode during analyzer calibration.
6. Sequencer operation with legend of binary codes.
7. Used for debugging purposes.
8. Used for debugging purposes.

Figure D8. Manual_02 Panel.

STABILITY

1. Updates the LabView RT application with the settings in (2), (3), (4), and (5).
2. This value represents the definition of "stable_flow". The flow is stable if the flow of each chamber is within + or - the percent value for the amount of time in (3).
3. The time that the flow of all chambers must be within the specified error percentage range in (2).
4. This value represents the definition of "stable_duct_temp". The temperature is stable if the temperature readings from sides 1 and 2 are within + or - the percent value for the amount of time in (5).
5. The time that the cabinet temperature must be within the percent error range (4).
6. Updates the RT application's sample line sequence with the host's.
7. This is the sequence in which gas sampling takes place. Sampling starts with the first entry and cycles back to the first when the end is reached. The sample lines must be either: one, two, three, four, five, six, inlet with no leading or trailing whitespace.

Figure D9. Stability Panel.

ERROR

1. Check this box if you want to enable the e-mail error notification system.
2. This list contains all the e-mail addresses that error messages will be sent to. To add more, click on the up arrow until you find a blank space. Then type in the address of the desired e-mail recipient.
3. This table is where the user can specify a low and high value for when error messages are issued. All ranges are labeled. If the value for the particular value goes out of range of the low and high values, the system is shut down to inactive mode and a critical error message is set.
4. Updates the RT (Real Time) application with the host's error range data. Since the RT application issues error messages, no changes will be seen until the button is pressed.
5. Display for the last LabView error code and a brief description of the error.
6. The mail server to be used to send e-mail notifications.

Figure D10. Error Panel.

WARNING

1. Specify the repeat interval of warning messages here. All values in seconds.
2. This table is where the user specifies the low and high values for when warning messages are issued. If a certain signal exceeds either boundary, a warning message about the error will be displayed on the front panel. See main.jpg (12).
3. Updates the RT application with the new warning information. Since the warning system is contained in the host application, these values aren't doing anything? Press the button when you make changes anyway.
4. Flipping this toggle switch to the "UP" position will suppress warning messages altogether.

Figure D11. Warning Panel.

DATA_LOG

1. Toggles whether or not data is being logged.
2. This is the path the user sets where they want the log file to be written. Note that the FULL PATH must be entered with the trailing back-slash.
3. This is the approximate interval that the system logs data to the file. The interval is accurate to ± 1 second.
4. Not implemented yet.
5. The IP address of the remote computer you wish to send your data files to.
6. The port of the remote computer that you will connect through.
7. Interval between log file sends. (43200 = twice per day)

Figure D12. Data_log panel.

HOST

1. Terminate the host application. This doesn't effect the RT application at all.
2. This button saves the entire host's settings and values in a file called scr_config. The save option can be handy so you don't have to re-configure everything when the host application is restarted. Only one configuration can be saved at once.
3. Press this button to load the previously saved settings from the scr_config file. Note that an error will be generated if the settings haven't already been previously saved.
4. This edit box contains the IP address of the RT application. VI server requires this in order to do reads and writes. Don't make changes to this unless you know what you are doing.
5. Port for VI Server. This should always be 3363.

State descriptions: T = Powered or ON, F = Unpowered or OFF

	inactive	idle	idle_flue	run	run_NH ₃
Gate Valve	F	F	T	T	T
Purge Valve	F	F	T	T	T
NH ₃ Valve	F	F	F	F	T
Soot Valve:	Only powered when sootblowing				
Flow control	F	F	F	T	T
Temp control	F	T	T	T	T
NH ₃ control	F	F	F	F	T
Gas sampling	F	F	F	T	T

Figure D13. Host control panel.

d. Summary of All Data that is Logged

Catalyst dps (6)

Power supplies: 30W, 90W, 120V, 240V (status)

Cabinet temps (4: 1st side, 1st side redundant, 2nd side, 2nd side redundant)

System entrance temp (duct temp)

System exit temperature

Temp before catalysts

Temperature after catalysts (6)

Chamber flow rates (6)

NH₃ supply pressure

Air Filter dp

Total dp

Measured NH₃ flowrate

Measured NO_x
Measured O₂
Current state of the control system
Control system target state
Time of last sootblowing
Current control valve outputs (6)
Current NH₃ Flowrate mfc (output)
Current value of cabinet heaters (output)
Valve status: Gate, soot, purge, NH₃
Current sample line

What can be controlled from the host PC?

Reactor state (Inactive, Idle, Idle flue, Run, Run with NH₃)

Setpoints:

Total flow rate (scfm)
Cabinet temperature (F)
NH₃ Stoichiometric ratio
Sample line sequence

Sootblowing:

Manual sootblowing event
Daily timed sootblow (Hour of day in military time)
dp triggered sootblow (critical chamber dp (H₂O))

Reset critical error: Reset the control system due to a critical error

Emergency shutdown: terminate the control system immediately

Gas Sampling:

Sample line sequence
Time to wait before sample is valid
Time to sample each chamber

Maintenance (Calibration):

ENABLED/DISABLED
Time for span gas to flow
Time for zero gas to flow

Data Logging:

File path
Interval between logs

Warning / Error ranges:

All High/low warning/error ranges

Warning repeat intervals
Suppression of warnings

E. SCR EQUIPMENT OPERATION AND INSPECTIONS

a. Suggested List of Equipment Checks

Here is the suggested list of checks to make on the equipment:

1. Check the CEMs cabinet for the following:
 - a. Check that the sample rotameters for NO_x and O₂ on the front panel show flow in the acceptable range (between the two blue lines).
 - b. Visual inspection of the “Mist Catcher” filter. Record color. If it turns yellow-brown from top to bottom, it needs to be changed. Spares located in the bottom of the cabinet.
 - c. Remove the circular filter and inspect. If it is dirty, replace it. Spares located in the bottom of the cabinet. When the filter is replaced, make sure that the grooved side of the o-ring faces out.
2. Check the SEQUENCER cabinet for the following:
 - a. Swing back the black panel and inspect the filter visually. Record color. Change the filter if it is discolored from top to bottom. Spares located in the bottom of the cabinet.
3. At the AIR BOX check the following:
 - a. Visual inspection of the filter on the incoming plant air line.
4. At the calibration gas cylinders located near the CEMs. Check pressures on all cylinders.
 - a. Notify REI if pressures fall below a set value as follows:
Nitrogen: ____250____ psig
O₂ Cal Gas: ____250____ psig
NO_x Cal Gas: ____250____ psig
5. Check differential pressure on the incoming plant air.
6. In the Fan Room. Check pressure on ammonia cylinders.
 - a. Notify REI if pressures fall below a set value as follows:
Ammonia: ____20____ psig

b. Ammonia Usage and System Maintenance

Ammonia Cylinder Changeover. The layout for ammonia cylinders and the manifold is given in Figure E1. The manifold can hold four cylinders. At any time, two cylinders are on standby and two in use. The procedure for changing cylinders is as follows. Please refer to the schematic on Figure E1 for terminology used to describe the valves.

- (1) Change the two cylinders in use at the same time when the ammonia pressure in both drops to 20psi.
- (2) Shut-off the cylinder valves and ***Cylinder Isolation Valves***.
- (3) Isolate the bank of cylinders to be removed by shutting off the ***Manifold Selector Valve***.
- (4) Bleed the residual ammonia in the line by opening the ***Vent Valve***.
- (5) Open the ***Cylinder Isolation Valve*** to bleed the residual ammonia trapped between the cylinder and the isolation valve.
- (6) Shut-off the ***Cylinder Isolation Valves*** and the ***Vent Valve***.
- (7) Disconnect the empty cylinders from the manifold.
- (8) Connect replacement cylinders.
- (9) Open cylinder valves and ***Cylinder Isolation Valves*** for the standby cylinders.
- (10) Open the ***Manifold Selector Valve*** for the standby cylinders.
- (11) Check for leaks.
- (12) Set the ammonia pressure in the transfer line to a desired value (less than 85psi).
- (13) Remove the empty cylinders using a cart and store in a cool place (less than 95°F).
- (14) Record the date and time of cylinder changing.

Safety Precautions During Cylinder Changeover. The person changing cylinders should wear a full-face respirator and should carry at least an 8-ounce water squirt bottle. Rubber gloves with thermal lining should be used. For clothing, a lightweight rubber suit is recommended. At the very least a long sleeve shirt and cotton coveralls should be worn. An additional backup person should be present. The additional backup person can be either A Tanner Industries personnel delivering the fresh ammonia cylinders or a designated person from the plant who has been trained in ammonia safety. These safety precautions should also be observed whenever an ammonia process line is broken for maintenance or any other reason.

The storage of empty ammonia cylinders is not as critical as that for full cylinders. However, it is prudent to treat empty cylinders with the same caution given to full cylinders. Temperature storage restrictions for full cylinders should also be applied to empty cylinders.

Figure E1. Schematic of the ammonia cylinder station and the manifold.

Ammonia Emergency Response.

A. First Aid

First Aid for Inhalation: Remove the victim from the contaminated area to fresh air while protecting yourself. If the victim is not breathing initiate artificial respiration and supply oxygen if needed. Keep victim warm and at rest. Seek medical attention; pulmonary injury may continue to evolve over 18 to 24 hours. If patient is conscious, the irritation of the throat may be relieved by water in the mouth. Seek medical attention immediately.

First Aid for Eye Contact: Remove victim from the source of contamination and take to nearest eye wash or shower. Immediately wipe away any excess chemical very gently and quickly. Wash the affected eye or eyes under slowly running water for 15 minutes or longer, making sure the eyelids are held wide apart and moved slowly in all directions. Get medical attention immediately.

First Aid for Skin Contact: Remove victim from source of contamination and take immediately to nearest shower or source of clean water. Immediately flush skin with

plenty of water for at least 15 minutes while removing contaminated clothing and shoes. While washing victim down taking care to protect eyes. Wash until the feeling of stickiness or soapiness disappears. This may take an hour or more. Get medical attention immediately. Wash clothing before reuse. Thoroughly clean shoes before reuse.

First Aid for Ingestion or Swallowing: Loosen tight clothing around the neck and waist. Flush mouth several times with cold water and spit out. Give victim 1 to 2 cups of milk. Do not induce vomiting. Do not give oils or attempt to neutralize with an acid. Do not give sodium bicarbonate or carbonated drinks. If vomiting occurs, keep the head lower than the hips to prevent vomit from entering the lungs.

B. Accidental Release Measures

Approach release from upwind. Ventilate area of leak or spill. Wear appropriate personal protective equipment. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Carefully neutralize spill with dilute HCl. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Use water spray to cool, absorb, and disperse vapors. Do not use combustible materials, such as sawdust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

Ammonia System Maintenance. The ammonia cylinders, manifold and transfer line should be visually inspected once every week. Pay particular attention to piping, joints and valves. These should be examined for signs of corrosion, damage wear and leaks.

An ammonia utilization spreadsheet is available to help estimate cylinder life and changeover schedule. Table E1 below gives an example of ammonia usage when the flue gas NO_x concentration is assumed to be 253 ppm.

Table E1. Ammonia Usage

Incoming NO _x concentration:	253	ppmv	Pipe diameter:	4.0	inch
Target NH ₃ /NO _x stoichiometry:	1.10	NH ₃ /NO _x	Gas velocity:	23	ft/s
NH ₃ flow:	1.675	liter/min			
NH ₃ flow:	0.063	scfm			
NH ₃ flow:	4.027	pounds/day	Gas density:	0.633105	kg/m ³
NH ₃ requirement per month:	2701	scf/month		0.039457	lb/ft ³
Cylinder diameter:	15	inch			
Cylinder height:	52	inch			
Cylinder capacity:	150	pounds			
Cylinder cost:		dollars			
Cylinder life:	37.3	days			
Cylinders required for 180 days:	4.8	cylinders			

c. Equipment Logsheets.

See below

CEMS AND SEQUENCER CHECKLIST

[illegible]

Note:

- (1) Filters in the CEMS's air conditioner and the Sequencer's vents (backdoor) must also be inspected once every two weeks.
- (2) Check ammonia cylinders weekly. Cylinder and line pressures not to exceed 125psi and 85psi respectively.
- (3) Cal gas tank pressures should be checked weekly.
- (4) Filters and flow meters in the CEMS and Sequencer should be inspected twice a week. Once a day for first week.
- (5) Highlight or note when any filter is changed.
- (6) Fax Log Sheets every week to: Connie Senior. Fax: (801) 364 6977
- (7) *Check if rotameter float is between blue lines.

CEMS AND SEQUENCER CHECKLIST

[illegible]

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CEMS AND SEQUENCER CHECKLIST

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CEMS AND SEQUENCER CHECKLIST

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- (5) Highlight or note when any filter is changed.
- (6) Fax Log Sheets every week to: Connie Senior. Fax: (801) 364 6977
- (7) *Check if rotameter float is between blue lines.

d. Notes on Equipment Checklist

AC Filters on CEMS and SEQUENCER. The AC filters on the CEMS and SEQUENCER need not be changed. However, if there is a dust buildup, the filters should be removed and the dust shaken off. If necessary, a blast of air may be applied to remove dust from the shaken filters after which the filters should be remounted on the equipment.

CONTACTS

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BALDWIN ENVIRONMENTAL		
		(775) 850 1800
TANNER INDUSTRIES		
Steve Stacey	Sales Rep	1-800-643-6226 EXT 491 (317) 341 0517 – for fast response
Jim Selby	Technical Support	1-800-643-6226 Ask for Jim
AMMONIA EMERGENCY		
TANNER INDUSTRIES		1-800-643-6226
CHEMTREC	Chemical Emergency Center	1-800-424-9300.

F. ANHYDROUS AMMONIA MATERIAL SAFETY DATA SHEET

MATERIAL SAFETY DATA SHEET																								
ANHYDROUS AMMONIA
DISTRIBUTORS: TANNER INDUSTRIES, INC.																						
DIVISIONS:																								
NATIONAL AMMONIA		NORTHEASTERN AMMONIA																						
HAMLER INDUSTRIES		BOWER AMMONIA & CHEMICAL																						
735 Davisville Road, Third Floor, Southampton, PA 18966; 215-322-1238 CORPORATE EMERGENCY TELEPHONE NUMBER: 800-643-6226; CHEMTREC (CMA) 800-424-9300																								
DESCRIPTION																								
CHEMICAL NAME: Ammonia, Anhydrous		CAS REGISTRY NO: 7664-41-7																						
SYNONYMS: Ammonia		FORMULA: NH ₃																						
CHEMICAL FAMILY: Ammonia		MOL. WT: 17.03 (NH ₃)																						
COMPOSITION: 99+% Ammonia																								
STATEMENT OF HEALTH HAZARD																								
HAZARD DESCRIPTION: Ammonia is an irritant and corrosive to the skin, eyes, respiratory tract and mucous membranes. May cause severe chemical burns to the eyes, lungs and skin. Skin and respiratory related diseases could be aggravated by exposure. Not recognized by OSHA as a carcinogen. Not listed in the National Toxicology Program. Not listed as a carcinogen by the International Agency for Research on Cancer.																								
EXPOSURE LIMITS FOR AMMONIA: Vapor <table style="width: 100%; margin-top: 5px;"> <tr> <td style="width: 30%;">OSHA</td> <td style="width: 20%;">50 ppm,</td> <td style="width: 20%;">35 mg / m³ PEL</td> <td style="width: 30%;">8 hour TWA</td> </tr> <tr> <td rowspan="3">NIOSH</td> <td>35 ppm,</td> <td>27 mg / m³ STEL</td> <td>15 minutes</td> </tr> <tr> <td>25 ppm,</td> <td>18 mg / m³ REL</td> <td>10 hour TWA</td> </tr> <tr> <td>300 ppm,</td> <td>IDLH</td> <td></td> </tr> <tr> <td rowspan="2">ACGIH</td> <td>25 ppm,</td> <td>18 mg / m³ TLV</td> <td>8 hour TWA</td> </tr> <tr> <td>35 ppm,</td> <td>27 mg / m³ STEL</td> <td>15 minutes</td> </tr> </table>				OSHA	50 ppm,	35 mg / m ³ PEL	8 hour TWA	NIOSH	35 ppm,	27 mg / m ³ STEL	15 minutes	25 ppm,	18 mg / m ³ REL	10 hour TWA	300 ppm,	IDLH		ACGIH	25 ppm,	18 mg / m ³ TLV	8 hour TWA	35 ppm,	27 mg / m ³ STEL	15 minutes
OSHA	50 ppm,	35 mg / m ³ PEL	8 hour TWA																					
NIOSH	35 ppm,	27 mg / m ³ STEL	15 minutes																					
	25 ppm,	18 mg / m ³ REL	10 hour TWA																					
	300 ppm,	IDLH																						
ACGIH	25 ppm,	18 mg / m ³ TLV	8 hour TWA																					
	35 ppm,	27 mg / m ³ STEL	15 minutes																					
TOXICITY: LD 50 (Oral / Rat) 350 mg / kg																								
PHYSICAL DATA																								
BOILING POINT: -28°F PH: N/A VAPOR DENSITY (air = 1): 0.596 at 32°F SPECIFIC GRAVITY (water = 1): 0.682 at 39°F PERCENT VOLATILE: 100% at 212°F APPEARANCE AND ODOR: Colorless liquid or gas with pungent odor. CRITICAL TEMPERATURE: 270.3°F		APPROXIMATE FREEZING POINT: -108°F WEIGHT (per gallon): 5.69 pounds at -28°F VAPOR PRESSURE: 10 atm at 78°F SOLUBILITY IN WATER: 100% EVAPORATION RATE (water = 1): Similar SURFACE TENSION: 23.4 Dynes / cm at 52°F CRITICAL PRESSURE: 111.5 atm																						
EMERGENCY TREATMENT																								
EFFECTS OF OVEREXPOSURE: <i>Eye:</i> Tearing, edema or blindness may occur. <i>Skin:</i> Irritation, corrosive burns, blister formation may result. Contact with liquid may produce a caustic burn and frostbite. <i>Inhalation:</i> Acute exposure may result in severe irritation of the respiratory tract, bronchospasm, pulmonary edema or respiratory arrest. <i>Ingestion:</i> Lung irritation and pulmonary edema may occur. Extreme exposure may result in death from spasm, inflammation or edema. Brief inhalation exposure to 5,000 ppm may be fatal.																								

Revision: May 2001

Page 1 of 4

Prepared By: JRW

MATERIAL SAFETY DATA SHEET**EMERGENCY AID:**

Eye: Flush with copious amounts of water for a minimum of 20 minutes. Eyelids should be held apart and away from eyeball for thorough rinsing.

Skin: Flush with copious amounts of water for a minimum of 20 minutes while removing contaminated clothing and shoes. Do not rub or apply ointment on affected area. Clothing may initially freeze to skin. Ensure clothing is not frozen prior to removal.

Inhalation: Remove to fresh air. Administer oxygen or artificial respiration, if necessary.

Ingestion: If conscious, give large amounts of water to drink. May drink orange juice, citrus juice or diluted vinegar (1:4) to counteract ammonia.

DO NOT INDUCE VOMITING! SEEK IMMEDIATE MEDICAL HELP FOR ALL EXPOSURES!

NOTE TO PHYSICIAN: Respiratory injury may appear as a delayed phenomenon. Pulmonary edema may follow chemical bronchitis. Supportive treatment with necessary ventilation actions, including oxygen, may warrant consideration.

FIRE AND EXPLOSION HAZARD DATA

FLASHPOINT: None

FLAMMABLE LIMITS IN AIR: LEL / UEL 16% to 25%

LEL / UEL listed in the *NIOSH Pocket Guide to Chemical*

Hazards as 15% to 28%

AUTO IGNITION TEMPERATURE: 1,204°F (If Catalyzed)

1,570°F (If Uncatalyzed)

EXTINGUISHING MEDIA: Non-Combustible

SPECIAL FIRE-FIGHTING PROCEDURES:

Must wear protective clothing and a positive pressure SCBA. Stop source if possible. Fight fires using dry chemical, carbon dioxide, or alcohol-resistant foam. Cool fire exposed containers with water spray. Stay upwind when containers are threatened. Use water spray to knock down vapor and dilute.

UNUSUAL FIRE AND EXPLOSION HAZARDS:

Outdoors, ammonia is not generally a fire hazard. Indoors, in confined areas, ammonia may be a fire hazard, especially if oil and other combustible materials are present. Combustion may form toxic nitrogen oxides.

If relief valves are inoperative, heat exposed storage containers may become explosion hazards.

CHEMICAL REACTIVITY**STABILITY:**

Stable at room temperature. Anhydrous Ammonia will react exothermically with acids and water.

CONDITIONS TO AVOID:

Avoid Anhydrous Ammonia contact with chemicals such as hydrocarbons, strong oxidizers, mercury, chlorine, iodine, bromine, silver oxide or hypochlorites; they can form explosive compounds.

Avoid Anhydrous Ammonia contact with chlorine, which forms a chloramine gas, which is a primary skin irritant and sensitizer.

Anhydrous Ammonia has a corrosive reaction with galvanized surfaces, copper, brass, bronze, aluminum alloys, mercury, gold and silver.

HAZARDOUS DECOMPOSITION PRODUCTS:

Anhydrous Ammonia decomposes to hydrogen and nitrogen gases above 450°C (842°F). Decomposition temperatures may be lowered by contact with certain metals.

SPILL OR LEAK PROCEDURES**STEPS TO BE TAKEN:**

Wear respiratory protection and protective clothing; see PROTECTIVE EQUIPMENT. Stop source if possible. Stay upwind and use water spray downwind of container to absorb the evolved gas. Contain spill and runoff from entering drains, sewers, and water systems by utilizing methods such as diking, containment, and absorption. CAUTION: ADDING WATER DIRECTLY TO LIQUID SPILLS WILL INCREASE VOLATILIZATION OF AMMONIA, THUS INCREASING THE POSSIBILITY OF EXPOSURE.

WASTE DISPOSAL:

Listed as hazardous substance under CWA (40 CFR 116.4, 40 CFR 117.3). Reportable Quantity 100 pounds. Classified as hazardous waste under RCRA (40CFR 261.32 Corrosive #D002). Comply with all regulations. Suitably diluted product may be disposed of on agricultural land as fertilizer. Keep spill from entering streams, lakes, or any water systems.

Revision: May 2001

Page 2 of 4

Prepared By: JRW

MATERIAL SAFETY DATA SHEET**SPECIAL PROTECTION AND PROCEDURES****RESPIRATORY PROTECTION:**

MSHA/NIOSH approved respiratory protection that consists of a full-face gas mask and canisters effective for ammonia that enable use for entry and escape in emergencies. Refer to 29 CFR 1910.134 and ANSI: Z88.2 for requirements and selection. A positive pressure SCBA is required for entry into ammonia atmospheres at or above 300 ppm (IDLH).

VENTILATION:

Local exhaust should be sufficient to keep ammonia vapor to 25 ppm or less.

PROTECTIVE EQUIPMENT:

Splash proof, chemical safety goggles, rubber gloves and boots should be worn to prevent contact. Face shield can be worn over the goggles as additional protection. Respiratory protection and cotton work clothes are recommended. Refer to 29 CFR 1910.132 through 1910.138 for requirements. A positive pressure SCBA is required for entry into ammonia atmospheres at or above 300 ppm (IDLH).

SPECIAL PRECAUTIONS**STORAGE AND HANDLING:**

Store in cool (26.7°C / 80°F) and well-ventilated area, with containers tightly closed. OSHA 29 CFR 1910.111 prescribes handling and storage requirements for anhydrous ammonia as a hazardous material.

WORK-PLACE PROTECTIVE EQUIPMENT:

Protective equipment should be stored near, but outside of anhydrous ammonia area. Water for first aid, such as an eyewash station and safety shower, should be kept available in the immediate vicinity. See 29 CFR 1910.111 for workplace requirements.

DISPOSAL:

See WASTE DISPOSAL. Classified as RCRA Hazardous Waste due to corrosivity with designation D002, if disposed of in original form.

LABELING AND SHIPPING

HAZARD CLASS: 2.2 (Non-Flammable Gas) US Domestic AND 2.3 (Poison Gas) International

PROPER SHIPPING DESCRIPTION: Ammonia, Anhydrous, 2.2, UN1005, RQ, Inhalation Hazard (US Domestic)
AND Ammonia, Anhydrous, 2.3, UN1005, RQ, Poison-Inhalation Hazard Zone "D" (International)

PLACARD: Non-Flammable Gas (US Domestic)

AND Poison Gas, Corrosive (Subsidiary) (International)

IDENTIFICATION NO: UN 1005

National Fire Protection Assoc. Hazardous Rating: **Hazardous Materials Identification System Labels:**

ANHYDROUS AMMONIA	
HEALTH	3
FLAMMABILITY	1
REACTIVITY	0
PERSONAL PROTECTION	H

MATERIAL SAFETY DATA SHEET**OTHER REGULATORY REQUIREMENTS**

Under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA), Section 103, any environmental release of this chemical equal to or over the reportable quantity of 100 lbs. must be reported promptly to the National Response Center, Washington, D.C. (1-800-424-8802).

The material is subject to the reporting requirements of Section 304, Section 312 and Section 313, Title III of the Superfund Amendments and Reauthorization Act of 1986 and 40 CFR 372. EPCRA extremely hazardous substance, 40 CFR 355, Title III, Section 302 – Ammonia, TPQ 500 lbs.

EPA Hazard Categories - Immediate: Yes; Delayed: No; Fire: No; Sudden Release: Yes; Reactive: No.

Clean Air Act – Section 112(r): Material is listed under 40 CFR 68.130 (EPA's Risk Management Program) at concentrations amounts greater than 10,000 lbs. (TQ).

DISCLAIMER

The information, data, and recommendations in this material safety data sheet relate only to the specific material designated herein and do not relate to use in combination with any other material or in any process. The information, data, and recommendations set forth herein are believed by us to be accurate. We make no warranties, either expressed or implied, with respect thereto and assume no liability in connection with any use of such information, data, and recommendations.

Revision: May 2001

Page 4 of 4

Prepared By: JRW