DOE/ DOE/PC/90361--T4 T4

DE92 005199

CATALYTIC FABRIC FILTRATION FOR SIMULTANEOUS NO_x AND PARTICULATE CONTROL

Quarterly Technical Progress Report
for the period July 1 - September 30, 1991

by

Greg F. Weber, Project Manager
Sumitra R. Ness, Research Engineer
Dennis L. Laudal, Research Engineer
Todd A. Svihovec, Graduate Research Assistant

Energy and Environmental Research Center
University of North Dakota
Box 8213, University Station
Grand Forks, ND 58202

Contracting Officer's Representative: Ms. Felixa Eskey

for

U.S. Department of Energy
Pittsburgh Energy Technology Center
626 Cochran Mill Road
Pittsburgh, PA 15236-0940

October 1991

MASTER

Work Performed Under Contract No. DE-AC22-90PC90361

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CATALYTIC FABRIC FILTRATION FOR SIMULTANEOUS NO_x AND PARTICULATE CONTROL

1.0 INTRODUCTION

The University of North Dakota Energy and Environmental Research Center (EERC), Owens-Corning Fiberglas Inc. (OCF), and Stearns-Roger, a division of United Engineers & Constructors (UE&C) have initiated a research project aimed at the development of a catalytic fabric filter for simultaneous NO_x and particulate control. Funding for the project is being provided by the U.S. Department of Energy/Pittsburgh Energy Technology Center (DOE/PETC), Consolidated Edison Company of New York Inc. (Con Edison), and the Empire State Electric Energy Research Corp. (ESEERCO).

The DOE/PETC funding was secured as a result of a competitive award from the DOE/PETC Advanced NO_x Control program. The objective of this program is to develop advanced concepts for removal of NO_x from flue gas emitted by coal-fired utility boilers, or for the control of NO_x formation by advanced combustion modification techniques. Funded projects are required to focus on the development of technology that significantly advances the state of the art using a process or a combination of processes capable of reducing NO_x emissions to 60 ppm or less. The concept must have successfully undergone sufficient laboratory-scale development to justify scaleup for further evaluation at the pilot scale (not to exceed 5 MWe in size). Other requirements include production of a nonhazardous waste or a saleable by-product. The concept should have application to both new and retrofit coal-fired systems or retrofit coal-fired systems. Also, the concept should show the potential for a 50% cost savings when compared to a commercial selective catalytic reduction (SCR) process capable of meeting the 60 ppm NO_x emission limit.

The EERC approach to meeting the program objective involves the development of a catalytic fabric filter for simultaneous NO_x and particulate control. The idea of applying either permanent or throwaway catalysts to a high-temperature fabric filter for NO_x control is not new (1-4). However, advances at OCF have shown that a high-activity catalyst can be applied to a high-temperature woven glass cloth resulting in a fabric filter material that can operate at temperatures higher than commercially available, coated glass fabric. In bench-scale experiments, over 90% of the NO_x is removed by catalytic reduction with ammonia to form nitrogen and water. The catalyst employed at this time is vanadium/titanium, but the exact catalyst composition and the unique method of applying the catalyst to high-temperature glass fabric are the property of OCF (5). Other catalyst options are being evaluated by OCF in order to improve catalyst performance and minimize catalyst cost.

Experimental results to date have shown that over 90% NO_x removal can be achieved, the catalyst/fabric has promising self-abrasion characteristics, and the potential exists for substantially reduced cost when compared with conventional SCR/fabric filtration technology (6,7). However, several aspects of the technology require optimization and/or further evaluation. These include air-to-cloth ratio, ammonia slip, SO_2 oxidation to SO_3, temperature cycling, catalyst-coated fabric preparation, fuel impacts, fabric cleaning (reverse-gas versus pulse-jet), catalyst life (poisoning and resistance to erosion), and filter performance/life (particulate control, differential pressure, and abrasion resistance).

The specific approach being used to address these issues is represented by the following:

Task 1 - Program Definition
Task 2 - Design and Construct Test Unit
Task 3 - Experimental Program and Data Reduction
Task 4 - Conceptual Design and Economic Evaluation
Task 5 - Test Unit Removal

Task 1, Program Definition, required preparation of a Project Management Plan. This document contains a detailed scope of work, project schedule, and project budget; identifies key project personnel; and presents an appropriate QA/QC plan for the project. Task 2, Design and Construct Test Unit, is divided into three subtasks. Subtasks 2.1 and 2.2 involve the detailed design of the test facility on which the experimental program will be performed, the construction of the test facility, and an operational shakedown of the system prior to initiating the experimental effort. Subtask 2.3 involves OCF preparation of the catalyst-coated fabric filters that will be evaluated in the experimental effort.

Task 3, Experimental Program and Data Reduction, has four subtasks. Subtask 3.1, Fundamental Testing, is intended as a support effort to the primary experimental test plan. This effort should supply information needed to thoroughly understand the process mechanism, allow screening of additional fabric samples, address the issue of catalyst poisoning, and generally support the pilot-scale effort. Subtask 3.2, Process Testing/Reverse-Gas System, is intended to evaluate process performance in a pilot-scale reverse-gas fabric filter, identify and optimize important process variables, and provide data for a conceptual design. Subtask 3.3, Process Testing/Pulse-Jet System, will evaluate process performance in a pilot-scale pulse-jet fabric filter. Subtask 3.4, Fabric Durability Testing/Pulse-Jet System, will evaluate catalyst-coated fabric durability/performance for up to 12,000 hours in a slipstream pulse-jet baghouse.

Based on the results of Task 3, a technical and economic assessment of the catalytic fabric filter concept will be completed in Task 4, Conceptual Design and Economic Evaluation. Task 5, Test Unit Removal, requires the identification of all major test facilities, equipment, instruments and hardware, and their condition, ownership, and proposed disposition.

EERC is the primary contractor for the project and is responsible for the management/direction of all technical and administrative project activities. EERC will specifically perform all work associated with Task 1, Subtasks 2.1 and 2.2, Task 3, and Task 5.

OCF will perform and/or supervise all work associated with Subtask 2.3, including manufacturing the "S" glass fabric, coating the fabric, and manufacturing the filter bags for the reverse-gas and pulse-jet systems. This effort, as well as technical support, will be provided by OCF as a cost share to the project.

UE&C, as a subcontractor to EERC, will provide technical support for Task 1 and will perform Task 4. EERC personnel involved in the Task 3 effort and OCF personnel will provide input to UE&C Task 4 activities.

The project schedule is based on a project start date of October 1, 1990, and assumes a project duration of 36 months. Therefore, the planned project completion date is September 30, 1993.

Federal funding accounts for roughly 56% of the total project budget. Commercial funding for the project makes up the remaining 44%. Con Edison and ESEERCO are each funding 12% of the project cost, and OCF is contributing 10% in the form of catalyst-coated filter bags and technical support for the project. The remaining 10% is uncommitted at this time, but EERC is pursuing funding options in an effort to obtain a formal commitment for the remaining commercial cost share as soon as possible.

2.0 GOALS AND OBJECTIVES

The overall objective of this project is the development of a catalytic fabric filter for simultaneous NO_x and particulate control. The catalytic fabric filter must provide high removal efficiency of NO_x and particulate matter. An acceptable bag and catalyst life must be demonstrated, and process economics must show a significant cost savings when compared to a commercial SCR process and conventional particulate control. Specific goals include the following:

- Reduce NO_x emissions to 60 ppm or less.
- Demonstrate particulate removal efficiency of >99.5%.
- Demonstrate a bag/catalyst life of >1 year.
- Control ammonia slip to <25 ppm.
- Show that catalytic fabric filtration can achieve a 50% cost savings over conventional fabric filtration and SCR control technology.
- Determine compatibility with SO_2 removal systems.
- Show that the concept results in a nonhazardous waste product.

Specific project activities during the past quarter (July through September 1991) were to include the following:

- Complete Task 2, Design and Construct Test Unit.
- Continue Subtask 3.1, Fundamental Testing.
- Select coals to be used during Task 3 pilot-scale activities.
- Initiate Subtask 3.2, Process Testing/Reverse-Gas System.

3.0 ACCOMPLISHMENTS

3.1 Task 2, Design and Construct Test Unit

Task 2, Design and Construct Test Unit, was initiated in January 1991 after DOE/PETC approved the Project Management Plan. Activities in the past quarter have focused on completing construction of and shakedown tests with the pilot-scale reverse-gas (Subtask 2.1) and pulse-jet (Subtask 2.2) baghouses.

Construction and assembly of the reverse-gas and pulse-jet baghouses were completed in August. The final as-built drawings and equipment lists for the reverse-gas and pulse-jet baghouses were prepared in conjunction with the construction effort. Most of this information was presented in the document entitled "Catalytic Fabric Filter Test Unit Design," attached to the Quarterly Technical Progress Report for the period April through June 1991. But due to the delays encountered with completion of the construction activities, several new and revised drawings have been prepared and are presented in an updated document attached to this report, entitled "Catalytic Fabric Filter Test Unit Design."

Shakedown tests with the reverse-gas and pulse-jet baghouses were initiated in August and completed in early September. The purpose of the shakedown tests was to verify mechanical operation of the new baghouses to assure that the operating conditions contained in the experimental work plan could be achieved.

Standard woven glass bags were installed in both the reverse-gas and pulse-jet baghouses for the shakedown tests. These bags were supplied by Owens-Corning Fiberglas Inc. (OCF) through Menardi-Criswell (a division of Hosokawa Micron International Inc.). The reverse-gas bags were 12 inches in diameter and 25 feet 9 inches long with six anticollapse rings. Each bag was attached to stainless steel thimbles at the top and bottom of the baghouse using stainless steel hose clamps and tensioned using a load cell and tension spring. Filtration area for each bag was 78.5 ft^2, resulting in a total filtration area of 157 ft^2.

Twelve pulse-jet bags, 6 inches in diameter and 8 feet 3 inches long, were installed using snap bands sewn in the top cuff to secure the bags to the tube sheet. Stainless steel wire cages were then inserted into the bags to provide support. Each pulse-jet bag provided a filtration area of 12.57 ft^2, resulting in 150.8 ft^2 of total filtration area.

Shakedown tests were initially performed while firing the particulate test combustor (PTC) with natural gas and then with a washed Illinois #6 bituminous coal. Table 1 presents a complete analysis of the coal (weight percent as-fired) and coal ash (weight percent as oxides). The coal appears to be characteristic of midwestern bituminous coals: high fixed carbon (67.1%) and heating value (11,239 Btu/lb), moderate ash (11.5) and sulfur (3.0), and low moisture (8.5%) content. The coal ash was characterized by high silica (51.6%), alumina (21.4%), and iron (15.4%) and low alkali content.

TABLE 1

Coal and Ash Analyses for the Washed Illinois #6 Fuel Used During the System Shakedown Tests

Coal Type	Washed Illinois #6 Bituminous
Proximate Analysis (%)	
Moisture	8.5
Volatile Matter	12.3
Fixed Carbon	67.1
Ash	11.5
Ultimate Analysis (%)	
Hydrogen	5.4
Carbon	62.8
Nitrogen	1.1
Sulfur	3.0
Oxygen (Diff.)	16.1
Ash	11.5
Heating Value (Btu/lb)	11,239
Percent as Oxides*	
SiO_2	51.6
Al_2O_3	21.4
Fe_2O_3	15.4
TiO_2	0.9
P_2O_5	0.2
CaO	3.6
MgO	1.3
Na_2O	1.0
K_2O	2.0
SO_3	2.6

* Weight percent concentrations of oxides are normalized values.

Initial shakedown tests with the reverse-gas chambers were generally successful. Flue gas flow rates of 111 and 147 scfm were demonstrated at baghouse inlet temperatures ranging from 500° to 650°F. All system instrumentation (thermocouples, weigh cells, and differential and static pressure cells) appeared to function properly. A reverse-gas chamber temperature of 750°F was not demonstrated due to moderate air leaks (about 10% of the flue gas flow rate) observed in the reverse-gas chambers and problems encountered with several heating elements. The air leaks were corrected (reduced to 3% or less) by installing new gasket material. The failed heating elements were determined to be defective and are presently being repaired by the vendor. Once the repaired heating elements have been installed, good temperature control in the range of 500° to 750°F should be available.

Shakedown tests with the new pulse-jet baghouse were successfully completed with only minor air leaks (3% or less) observed. The flue gas flow rate was 141 scfm with baghouse inlet temperatures ranging from 500° to 650°F. Air-to-cloth ratios of 2, 3, 4, and 6 ft/min were demonstrated by plugging the outlets of individual bags while the baghouse was on-line. Although a baghouse inlet temperature of 750°F was not specifically demonstrated during the shakedown tests, it was demonstrated with a similar equipment configuration during bench-scale catalyst-coated fabric-screening tests performed on several occasions in the past two years.

Particulate sampling was satisfactorily performed during shakedown tests using existing sample port locations. The reverse-gas filter bags were effectively cleaned using a valve arrangement that diverts flue gas through the pulse-jet baghouse first to remove particulate and then passes it through the reverse-gas chambers from the clean-side to the dirty-side, partially collapsing the bags and dislodging the dust cake.

Tables 2 and 3 summarize the EPA Method 5 and multicyclone particulate sampling data for both the reverse-gas and pulse-jet baghouse shakedown tests. Based on two measurements, the particulate mass loading in the flue gas from the pilot-scale pulverized coal-fired combustor ranged from 3.4 to 3.8 gr/scf.

TABLE 2

Particulate Sampling Data From Shakedown Tests

Test Date[a]	Test Type	Inlet gr/scf	Outlet gr/scf	% H_2O	% Isokinetic	% Efficiency
08-28-91	EPA-5	3.47744	---	10.5	92.5	---
08-28-91	EPA-5	---	0.00151	9.1	89.9	99.96
08-28-91	FS#325	2.66107	---	10.2	97.0	---
08-29-91	EPA-5	3.78776	---	8.1	91.7	---
08-29-91[b]	EPA-5	---	0.04719	7.3	97.9	98.8
08-29-91	FS#326	2.54104	---	7.1	94.7	---

[a] Particulate sampling for the pulse-jet and reverse-gas chambers were performed on 08-28-91 and 08-29-91, respectively.

[b] The outlet grain loading is a result of the weight gain determined from the acetone wash of the probe, since no weight gain was observed on the filter. Therefore, EERC attributes the high outlet grain loading to construction residue in the baghouse outlet pipe, rather than fly ash penetration.

TABLE 3

Multicyclone Data From Shakedown Tests

08-28-91	CY1	CY2	CY3	CY4	CY5	Backup Filter
D_{50} (μm)	8.23	3.97	2.86	1.29	0.73	---
Mass Collected (g)	2.06740	0.23550	0.12570	0.04990	0.02460	0.00624
% of Total Mass	82.39	9.38	5.01	1.99	0.98	0.25
Cumulative % Mass Less than D_{50}	17.61	8.23	3.22	1.23	0.25	---

Total Mass Collected = 2.50934 g, Dust Loading = 2.66107 gr/scf
% Isokinetic = 97.0

08-29-91	CY1	CY2	CY3	CY4	CY5	Backup Filter
D_{50} (μm)	9.66	4.97	3.69	1.70	0.95	---
Mass Collected (g)	1.45780	0.17130	0.20000	0.04600	0.03260	0.00693
% of Total Mass	76.14	8.95	10.45	2.40	1.70	0.36
Cumulative % Mass Less than D_{50}	23.86	14.91	4.47	2.06	0.36	---

Total Mass Collected = 1.91463 g, Dust Loading = 2.54104 gr/scf
% Isokinetic = 94.7

The outlet loading from the pulse-jet baghouse was observed to be 0.00151 gr/scf, resulting in a collection efficiency of 99.96% This was somewhat less than the 99.99% anticipated. Upon completion of the shakedown tests and inspection of the pulse-jet baghouse, one bag was found to have a hole in it, possibly explaining the relatively low particulate collection efficiency observed. The hole was caused by a bent thermocouple that punctured the bag. Careful attention to thermocouple locations during bag installation should prevent this from happening in the future.

A calculated particulate collection efficiency of only 98.8% was determined for the reverse-gas baghouse based on a measure outlet loading of 0.04719 gr/scf. A review of the sampling data showed that no weight gain was observed on the filter of the particulate sample train, but a significant weight gain was observed in the acetone wash of the probe assembly. EERC personnel attribute this weight gain to particulate residue from construction of the reverse-gas baghouse outlet pipe, rather than fly ash penetration through the reverse-gas bags. Another possibility could be contaminated acetone used to wash the probe. But contaminated acetone is not likely since a similar result was not observed for the pulse-jet data. Particulate collection efficiency for the reverse-gas bags are expected to meet or exceed 99.99%.

Multicyclone sampling at the inlet of each baghouse was completed in order to determine the aerodynamic particle size of the fly ash in the flue gas stream entering the baghouses. Resulting data show that, for the pulse-jet baghouse, 82% of the mass had a particle size of greater than 8.23 μm. For the reverse-gas baghouse, the results were similar with 76% of the mass having a particle size greater than 9.66 μm. Mass loadings, as determined by multicyclone sampling, were roughly 1 gr/scf less than those determined by EPA Method 5. EERC experience has found that mass loading values from multicyclone sampling do vary somewhat from EPA Method 5 data. Therefore, during the 100-hour test periods, fly ash collected in the baghouses will be weighed in order to confirm sampling results. Also when collection efficiency is calculated, an average inlet mass loading will be used. This average inlet mass loading will be based on all the inlet EPA Method 5 sampling periods completed during a 100-hour test period.

Real-time particulate sampling was performed at the outlet of each baghouse using a TSI Aerodynamic Particle Sizer (APS-33) during the shakedown tests. The APS-33 laser particle sizer, manufactured by TSI Inc., can count and size particles in the 0.5- to 30-μm range. The primary advantages of this system are the high resolution and short sampling time. In the APS, particle-laden air is passed through a thin-walled orifice with the particles lagging behind the gas because of their higher inertia. The velocity lag is related to the aerodynamic diameter of the particles allowing the determination of the aerodynamic diameter of a particle by measuring the velocity of a particle as it exits from the orifice.

To measure the particle velocity, the APS employs a laser which is split into two beams and focused into two rectangular planes in front of the orifice. The light scattered by a particle passing though these beams is collected and focused onto a photomultiplier tube which emits two pulses separated by the time taken for the particle to cross the distance between the two planes. This time interval is measured electronically and used to calculate the particle's aerodynamic diameter. The APS consists of two main components: the sensor and the microcomputer system. The sensor module consists of an accelerating orifice and a laser velocimeter.

The purpose of sampling with the APS system was to generate flue gas particulate concentration data as a function of time downstream of each baghouse. Figures 1 and 2 illustrate the results. The APS data in Figure 1 show particulate emissions from the reverse-gas baghouse decreasing from 50 mg/m^3 (0.022 gr/scf) to 0.0001 mg/m^3 (0.000000044 gr/scf), as the initial dust cake was formed, and then stabilizing at about 0.005 mg/m^3 (0.0000022 gr/scf) after five hours. The emissions spike as a result of the cleaning cycle was roughly 10 mg/m^3 (0.0044 gr/scf). These data support the conclusion that the outlet emissions from the reverse-gas baghouse during the shakedown tests were significantly lower than the EPA Method 5 data indicated.

APS data for the pulse-jet baghouse, presented in Figure 2, shows outlet emissions ranging between 0.1 and 0.5 mg/m^3 (0.00004 and 0.00022 gr/scf) between cleaning cycles and spiking at roughly 10 mg/m^3 (0.0044 gr/scf) during each cleaning cycle. This data corresponds reasonably well to the EPA Method 5 measured outlet emission rate of 0.00151 gr/scf.

Bulk ash samples collected from the hoppers of the reverse-gas and pulse-jet baghouses were analyzed for carbon, major elements, and vanadium content. Table 4 presents carbon and major element content resulting from loss-on-ignition and x-ray fluorescence (XRF) analysis , respectively. The carbon content of the two samples averaged 4% by weight. Major ash constituents were silica, alumina, and iron, reported as weight percent as oxides.

Table 5 presents the vanadium and titanium content of the coal, coal ash, reverse-gas hopper ash, and pulse-jet hopper ash. The purpose of this analysis was to establish a baseline for future comparison with ash samples generated during testing of catalyst-coated bags. If significant levels of vanadium are lost from the surface of the bags, increased vanadium levels should be observed in the ash samples. A combination of XRF and atomic absorption (AA) analysis were used to make the determinations. The XRF and AA data agree reasonably well for the coal and three ash samples. Vanadium content of the coal and ash appears to be roughly 30 ppm and 200 to 250 ppm, respectively. Filter samples from EPA Method 5 outlet mass loading measurements have also been submitted for vanadium analysis, but the results were not available for inclusion in this report.

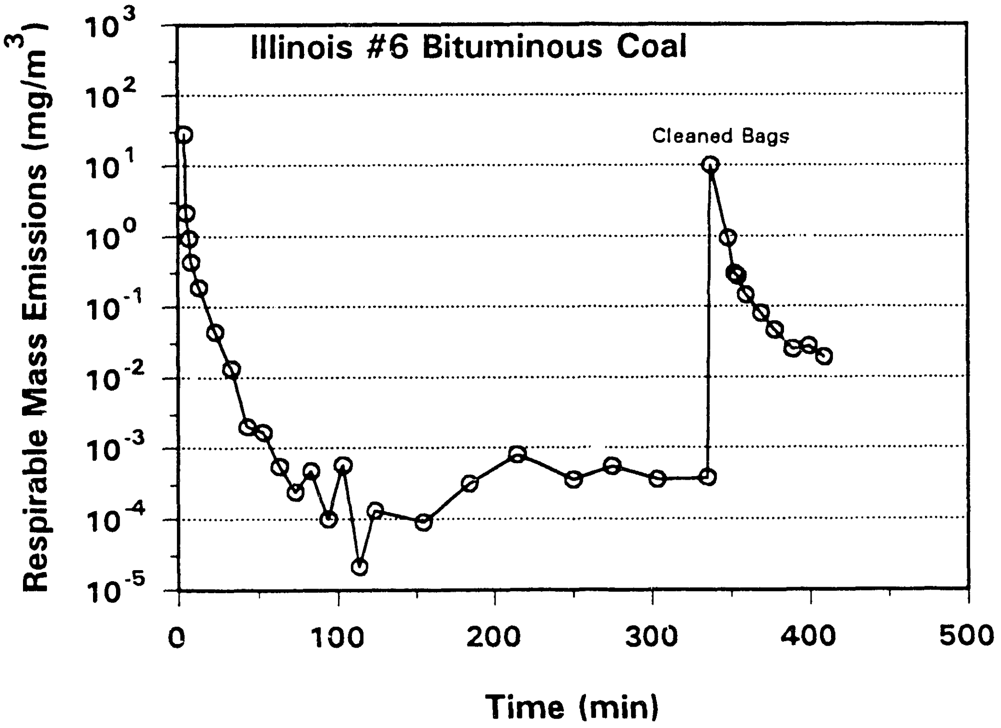

Figure 1. Respirable mass emissions versus time for reverse-gas shakedown tests.

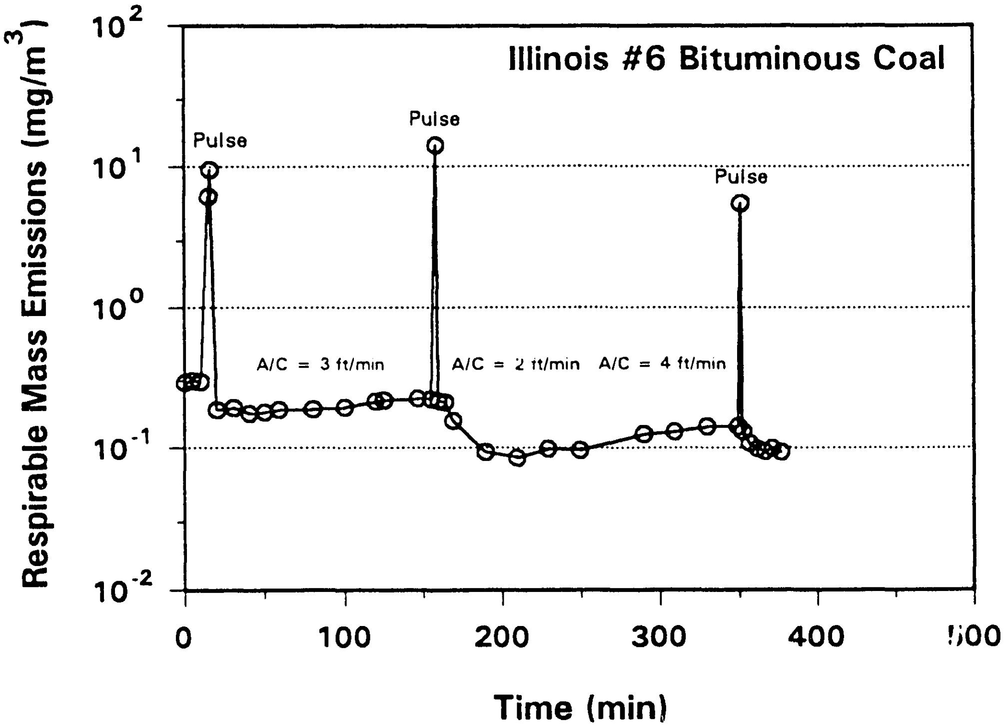

Figure 2. Respirable mass emissions versus time for pulse-jet shakedown tests.

TABLE 4

Ash Analysis for Samples Collected During System Shakedown Tests

	Pulse-Jet Baghouse Ash	Reverse-Gas Baghouse Ash
H_2O	---	---
Loss-On-Ignition	4.3	3.8
Percent as Oxides (wt%)	51.5	50.5
SiO_2	20.4	19.7
Al_2O_3	12.6	14.2
Fe_2O_3	0.9	0.8
TiO_2	0.1	0.2
P_2O_5	4.0	4.2
CaO	1.1	1.0
MgO	1.0	1.1
Na_2O	1.9	1.9
K_2O	2.2	2.6
SO_3		

TABLE 5

Vanadium and Titanium Concentrations for Samples Collected During System Shakedown Tests*

	Vanadium		Titanium	
	XRF, ppm	AA, ppm	XRF, ppm	AA, ppm
Washed Illinois #6 Bituminous Coal	28	31	590	570
Coal Ash	240	---	5160	---
Reverse-Gas Baghouse Ash	200	240	4800	5200
Pulse-Jet Baghouse Ash	210	250	5400	5500

* The vanadium and titanium content of the coal, based on XRF, was calculated from the XRF coal ash analysis and the ash content of coal.

Particle-size analysis of the bulk ash from the two baghouse ash hoppers was completed using a Coulter counter technique. Results, presented in Figure 3, show that the mass mean diameter (MMD) for the ash ranged from 13 to 20 μm. In order to get a complete particle-size distribution for the fly ash, it will be necessary to combine multicyclone and Coulter counter data. But since the basis for the measurements is different, multicyclone aerodynamic diameters versus Coulter counter volumetric diameters, Coulter counter data will have to be converted to an aerodynamic basis so it can be combined with multicyclone data, resulting in a complete particle-size distribution for fly ash entering the particulate control device. Particle-size distribution data resulting from the 100- and 500-hour experiments will be presented in this manner.

The on-line ammonia analyzer ordered from Siemens Energy & Automation, Inc. arrived in July. Unfortunately, setup and operating manuals for the instrument and the titanium dioxide converter were not included. The titanium dioxide converter and some setup information were obtained in August, but discussions with the vendor have been ongoing due to the limited documentation provided. Setup of the instrument was not completed in September as planned due to the delayed shipment of a portable mounting rack. As of the preparation of this report, the portable mounting rack is ready, and instrument setup should be completed in October.

The sulfur dioxide adsorbent was not shipped with the instrument in July and has not arrived as of the preparation of this report. Recent discussions with the vendor have determined that the original shipment was lost in transit and a second shipment, from Germany, should arrive in late October.

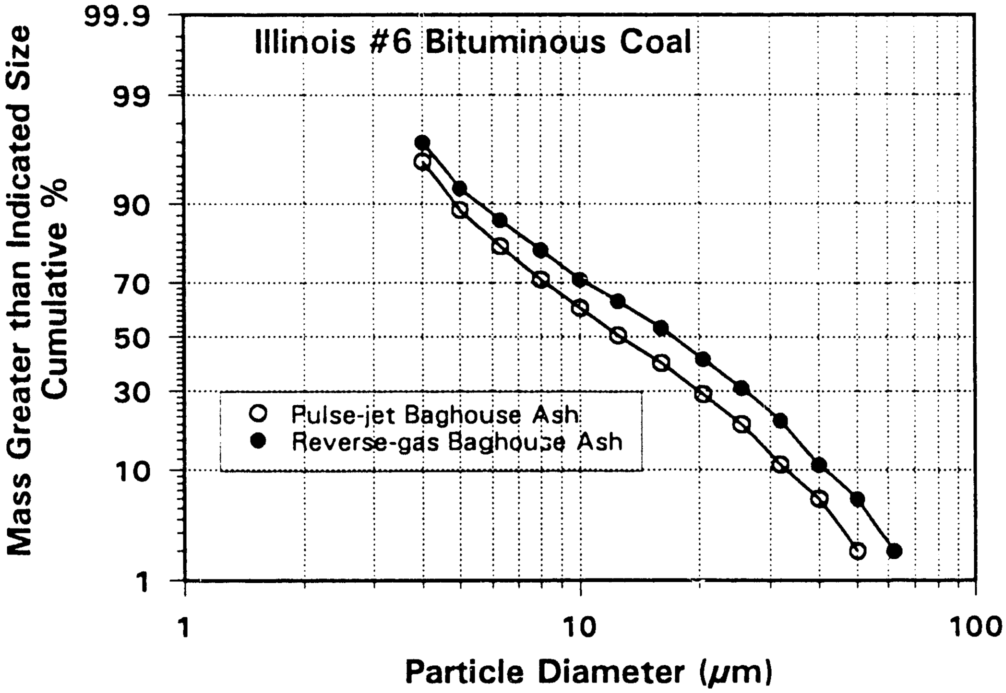

Figure 3. Coulter counter data for baghouse ash samples collected during system shakedown tests.

The reactor for the sulfur dioxide adsorbent has been constructed and tested using sodium carbonate as a possible replacement for the commercial adsorbent. The purpose of this test was to determine reactor capacity for sulfur dioxide control prior to tests with the ammonia analyzer in order to determine the recharging frequency that will be required during the week-long test periods.

Granular sodium carbonate was placed into the reactor, and the pressure drop was found to be 7-inches W.C. at ambient conditions and an air flow rate of 2 L/min. Subsequently, the reactor was electrically heated to 600°F, and a sulfur dioxide calibration gas was injected into the reactor inlet at 2 L/min. The sulfur dioxide concentration in the calibration gas was 1127 ppm with a nitrogen balance. An on-line sulfur dioxide analyzer was used to measure the sulfur dioxide concentration of the gas stream exiting the reactor.

For the first 70 minutes, the outlet sulfur dioxide concentration remained at zero; however, at about 75 minutes, there was a step change in the outlet sulfur dioxide concentration to 95 ppm. The calculated residence time for the empty reactor was only 30 seconds; therefore, this step change represents sulfur dioxide breakthrough or an analyzer malfunction. The outlet sulfur dioxide concentration then remained between 90 and 100 ppm for an additional 90 minutes until sulfur dioxide breakthrough began to increase. After 210 minutes, the sulfur dioxide concentration at the outlet of the reactor was 475 ppm. Based on these preliminary results, sodium carbonate may work as an adsorbing medium for short-term tests, but for longer-term testing, its use appears impractical. Plans to test sodium bicarbonate were not completed due to anticipated pressure drop problems in the reactor due to the small particle size of the material. EERC will continue to investigate other adsorbent options as an alternative to the Siemens sulfur dioxide adsorbent due the difficulty encountered in acquiring the Siemens material.

3.2 Subtask 3.1, Fundamental Testing

Shakedown tests with the gas chromatograph performed in early July showed that the instrument would not adequately measure nitric oxide or ammonia concentration. The problem was believed to be with the thermoconductivity detector, since component separation in the new column appeared adequate. Due to the age of the gas chromatograph, repair or replacement of the thermoconductivity detector was expected to be time consuming and/or expensive. In order to minimize further delays, EERC personnel elected to make use of an available Thermo Electron Model 10 NO_x analyzer for both nitric oxide (NO) and ammonia slip measurements. The instrument can be used in this manner as a result of its ability to measure both NO and total NO_x separately. When operated in the NO measurement mode, other nitrogen species such as nitrogen dioxide (NO_2) and ammonia (NH_3) are ignored. When operated in the total NO_x measurement mode, NH_3 is oxidized in the instrument's thermal converter and registers as NO. By switching between the two measurement modes, NH_3 concentration can be determined by difference. A colorimetric NH_3 measurement technique (Dragger tubes) was also used as a backup to the Thermo Electron Model 10 NO_x analyzer.

The proposed test matrix for the initial bench-scale experiments is summarized in Table 6. The primary variable to be evaluated during the first experimental series was air-to-cloth ratio (2, 3, 4, and 6 ft/min). Secondary variables included flue gas temperature (450°, 500°, 550°, 600°, 650°, 700°, and 750°F) and ammonia/nitric oxide molar ratio (0.8, 0.9, and 1.0). The shakedown tests were performed using the baseline test conditions and a new sample of the original Fabric #2.

The experimental apparatus, shown in Figure 4, consists of mass flow controllers, a tube furnace, a quartz reactor, a condenser, a temperature-controlled water bath, and a Thermo Electron NO_x chemiluminescence analyzer. Catalyst-coated fabric samples are loaded into the quartz reactor followed by an 18-mm ID glass tube to provide uniform gas flow through the fabric sample. Mass flow controllers are used to regulate composition and the flow rate of simulated flue gas. Simulated flue gas constituents include ammonia (NH_3), nitric oxide (NO), oxygen (O_2), and nitrogen (N_2). Temperatures are monitored by thermocouples along the furnace and near the surface of the fabric sample.

Individual component flow rates are set to attain a desired gas composition and total flow rate. In order to achieve a specific air-to-cloth ratio, the reactor is loaded with three equally cut fabric samples measuring approximately 22 mm in diameter. This results in a fabric surface area larger than the cross section of the reactor, allowing the system to operate at higher gas flow rates. An inner quartz tube, 18-mm ID, is inserted into the reactor and pushed up against the fabric surface, providing uniform gas flow through the fabric sample and quartz frit support.

TABLE 6

Test Matrix for Subtask 3.1--Fundamental Testing

Baseline Test Conditions

Inlet NO_x Concentration = 1000 ppm
NH_3/NO_x Molar Ratio = 0.9 (secondary variable)
Temperature = 650°F (secondary variable)
O_2 Concentration = 4.0%
Air-to-Cloth Ratio = 2 ft/min (primary variable)

Shakedown tests will be made using the baseline test conditions.

<u>Test Matrix A</u>

Air-to-Cloth Ratio (ft/min)	2	3	4	6	2	2
NH_3/NO_x Molar Ratio	0.9	0.9	0.9	0.9	0.9	0.9
Temperature (°F)	650	650	650	650	550	700

Fabrics #2, #17, #18, and #19 will be evaluated using Test Matrix A.

<u>Test Matrix B</u>

Air-to-Cloth Ratio (ft/min)	2	2	2	2	2	2
NH_3/NO_x Molar Ratio	0.9	0.9	0.9	0.9	0.9	0.9
Temperature (°F)	450	500	550	600	700	750

Fabric #18 will be evaluated using Test Matrix B.

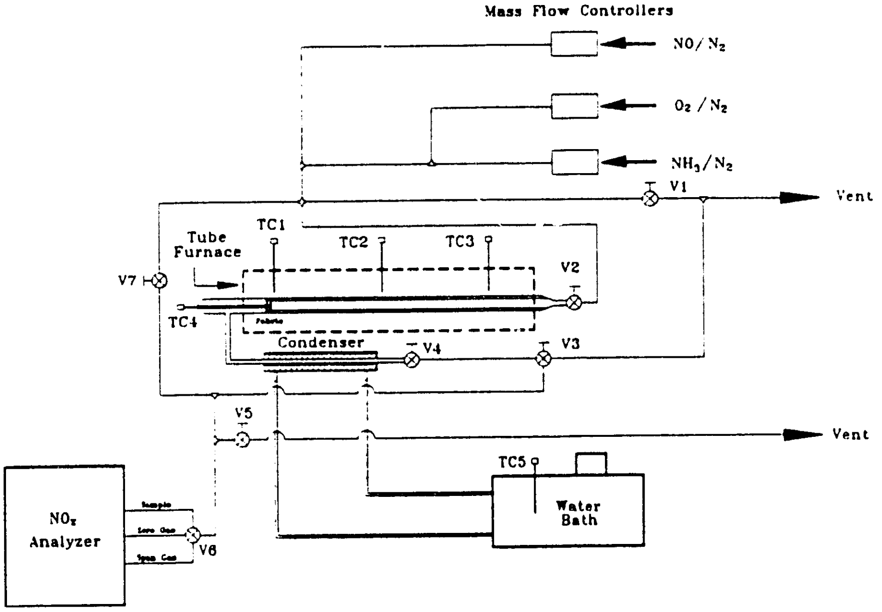

Figure 4. Bench-scale reactor system and NO_x analyzer.

Mass flow controllers were calibrated, and mass balance calculations were performed using a simultaneous equation solver software package. Table 7 illustrates the equations, required inputs, and resulting outputs. A bubble flowmeter was used for flow rate calibration due to the low flow rates required, and additional adjustments were necessary to correct for the solubility of ammonia and nitric oxide in water. The solubility issue was solved by allowing the respective gases to saturate the liquid. Flow rate calibration measurements were made at several different flowmeter settings (percent of full scale). After completing the calibration measurements, it became apparent that the mass flowmeters could not be easily adjusted to achieve NH_3/NO molar ratios of 0.8 and 1.0. Therefore, a NH_3/NO molar ratio of 0.9 was selected for all bench-scale screening experiments. A detailed discussion of the experimental procedure is presented in Appendix A.

Bench-scale experiments were initiated using a new sample of Fabric #2 (OCF-107) in order to evaluate the bench-scale experimental system and compare results with previous bench-scale fabric-screening activities (6,7). These initial experimental results compared favorably with the previous bench-scale

TABLE 7

Simultaneous Equations Solver
Mass Balance

Input	Name	Output	Unit	Comment
675	Total		cc/min	A/C ratio = 2 ft/min
0.033	x		% NO	Temperature = 650°F
0.0198	y		% NH_3	NH_3/NO molar ratio = 0.9
	A	20.45	cc/min	
	B	30.68	cc/min	
	C	623.86	cc/min	
0.0401	z	---	---	
	O_2 conc.	0.037	% O_2	
	Flow C	6	% scale	
	Flow B	11.7	% scale	
	Flow A	22.83	% scale	

Balance

(overall) Total = A + B + C
(NO) 0.001(Total) = x(A)
(NH_3) 0.0009(Total)= y(B)
(N_2) Total - x(A) - y(B) - z(C) = (1-x)A + (1-y)B + (1-z)C

Calibration Curves

(NO) Flow A = (A-0.7)/0.865
(NH_3) Flow B = (B+0.61)/2.68
(O_2) Flow C = (C-146)/80

results. Reduction of NO was in the range of 75% to 92% for air-to-cloth ratios of 6 and 2 ft/min, respectively. Ammonia slip was also found to be similar, ranging from about 20 to 140 ppm. Based on these results, screening tests with additional fabric samples were initiated.

The approach to the screening tests involved two steps. First, two swatches of each fabric type were evaluated individually at the baseline conditions listed in Table 6. The purpose for testing two swatches of each fabric type was to determine if any variability in the fabric samples existed. Once the initial screening test at baseline conditions was completed, those fabric types that demonstrated an NO_x reduction efficiency of 85% or better were evaluated using the composite experimental design presented in Table 8. This composite experimental design permits statistical analysis of the data and should result in a better understanding of potential fabric performance.

TABLE 8

Composite Experimental Design

X1 = Air-to-Cloth Ratio
X2 = Temperature

Run	X1	X2
1	-1	0
2	0	0
3	0	0
4	-1	-1
5	1	-1
6	-1	1
7	1	1
8	0	-alpha
9	0	alpha
10	-1	0
11	1	0
12	0	0
13	0	0

A/C
-1 = 2 ft/min
0 = 4 ft/min
1 = 6 ft/min

Temp
-1 = 550°F
0 = 650°F
1 = 750°F

-1.41 = 510°F
1.41 = 790°F

Alpha = $2^{0.5}$ = 1.41

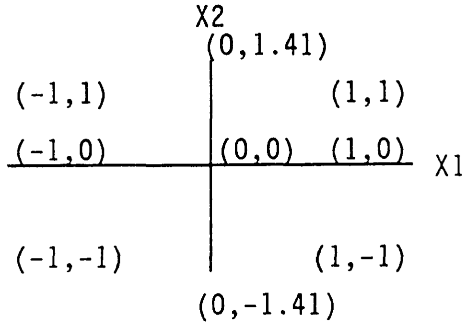

Table 9 presents the fabric samples prepared by OCF for screening with the bench-scale system and the results available as of the preparation of this report. The data for each fabric sample in Table 9 represents a minimum steady-state period of at least 30 minutes.

TABLE 9

Results of Bench-Scale Screening Tests

OCF-106A (Fabric #17, New V/Ti Catalyst)

Air/Cloth Ratio	2 ft/min	3 ft/min	4 ft/min	6 ft/min	2 ft/min	2 ft/min
Temperature (avg)	654°F	649°F	648°F	651°F	554°F	729°F
Length of Run	90 min	90 min	90 min	90 min	90 min	60 min
NH_3 Slip (avg)	63 ppm	56 ppm	121 ppm	171 ppm	110 ppm	24 ppm
NO_x Removal Eff. (%)	89.5	83.8	75.8	64.5	78.2	89.5
2 Std Dev.	±0.219	±0.153	±0.427	±0.333	±0.340	±0.194
C.V. (%)	±0.122	±0.091	±0.282	±0.258	±0.217	±0.109

OCF-107 (Fabric #2, Traditional V/Ti)

Air/Cloth Ratio	2 ft/min	3 ft/min	4 ft/min	6 ft/min	2 ft/min	2 ft/min
Temperature (avg)	655°F	658°F	647°F	655°F	546°F	703°F
Length of Run	90 min	90 min	90 min	90 min	90 min	90 min
NH_3 Slip (avg)	21 ppm	46 ppm	76 ppm	136 ppm	107 ppm	14 ppm
NO_x Removal Eff. (%)	92.7	86.0	82.4	74.7	80.9	94.4
2 Std Dev.	±0.163	±0.281	±0.407	±3.575	±0.761	±0.073
C.V. (%)	±0.088	±0.163	±0.247	±2.392	±0.471	±0.039

OCF-109 (Aqueous-Based V/Ti)

Air/Cloth Ratio	2 ft/min	Repeat 2 ft/min
Temperature (avg)	647°F	652°F
Length of Run	50 min	50 min
NH_3 Slip (avg)	283 ppm	358 ppm
NO_x Removal Eff. (%)	60.0	58.1
2 Std Dev.	±1.265	±0.872
C.V. (%)	±1.054	±0.751

OCF-140 (Aqueous-Based V/Ti)

Air/Cloth Ratio	2 ft/min	Repeat 2 ft/min
Temperature (avg)	649°F	652°F
Length of Run	30 min	30 min
NH_3 Slip (avg)	310 ppm	360 ppm
NO_x Removal Eff. (%)	54.5	55.5
2 Std Dev.	±2.380	±1
C.V. (%)	±2.184	±0.9009

continued...

TABLE 9 (continued)

Results of Bench-Scale Screening Tests

OCF-142 (Aqueous-Based V/Ti)

Air/Cloth Ratio	2 ft/min	Repeat 2 ft/min
Temperature (avg)	651°F	652°F
Length of Run	30 min	30 min
NH_3 Slip (avg)	45 ppm	65 ppm
NO_x Removal Eff. (%)	86.4	85.5
2 Std Dev.	±0.115	±0.000
C.V. (%)	±0.067	±0.000

OCF-143 (Aqueous-Based V/Ti)

Air/Cloth Ratio	2 ft/min	Repeat 2 ft/min
Temperature (avg)	652°F	649°F
Length of Run	40 min	30 min
NH_3 Slip (avg)	25 ppm	36 ppm
NO_x Removal Eff. (%)	92.8	90.5
2 Std Dev.	±0.049	±0.082
C.V. (%)	±0.026	±0.045

OCF-144 (Aqueous-Based V/Ti)

Air/Cloth Ratio	2 ft/min	Repeat 2 ft/min
Temperature (avg)	653°F	647°F
Length of Run	40 min	30 min
NH_3 Slip (avg)	104 ppm	85 ppm
NO_x Removal Eff. (%)	84.2	84.5
2 Std Dev.	±0.147	±0.000
C.V. (%)	±0.087	±0.000

OCF-145 (Fe/Ti Catalyst)

Air/Cloth Ratio	2 ft/min	Repeat 2 ft/min
Temperature (avg)	649°F	652°F
Length of Run	30 min	30 min
NH_3 Slip (avg)	333 ppm	380 ppm
NO_x Removal Eff. (%)	58.0	62.0
2 Std Dev.	±0.000	±0.000
C.V. (%)	±0.000	±0.000

OCF-117 (Fabric #18, Fe/Ti Catalyst)

Air/Cloth Ratio	2 ft/min
Temperature (avg)	649°F
Length of Run	30 min
NH_3 Slip (avg)	333 ppm
NO_x Removal Eff. (%)	58.0
2 Std Dev.	±0.000
C.V. (%)	±0.000

Data presented in Table 9 show that both OCF-107 (Fabric #2) and OCF-106A (Fabric #17) continue to demonstrate 90% NO reduction at an air-to-cloth ratio of 2 ft/min. In addition, a new fabric prepared using an aqueous-based vanadium/titanium catalyst also demonstrated >90% NO reduction at an air-to-cloth ratio of 2 ft/min. Based on these and previous results, OCF-107 (Fabric #2), OCF-106A (Fabric #17), and OCF-143 will be evaluated during the initial 100-hour pilot-scale test periods planned for November and December.

Although the initial screening test results with the iron/titanium-coated fabric samples only demonstrated 50% to 60% NO reduction, further bench-scale screening experiments will be performed as originally proposed (Table 6, Test Matrix B) to determine if NO reduction can be improved as a result of modified operating temperature.

Three fabric samples will also be evaluated using a Box-Behnken experimental design. Table 10 presents the Box-Behnken design, identifying the temperatures (550°, 650°, and 750°F) and the air-to-cloth ratios (2, 4, and 6 ft/min) to be used. Conditions have been chosen that will allow for an empirical method of analysis for measuring fabric performance. The initial

TABLE 10

Box-Behnken Experimental Design

X1 = Fabric
X2 = Temperature
X3 = Air-to-Cloth Ratio

Run	X1	X2	X3
1	-1	-1	0
2	-1	1	0
3	-1	0	-1
4	-1	0	1
5	1	0	1
6	1	0	-1
7	1	1	0
8	1	-1	0
9	0	0	0
10	0	-1	-1
11	0	-1	1
12	0	0	0
13	0	1	1
14	0	1	-1
15	0	0	0
Additional Tests			
16	-1	-2	-1
17	-1	-1.5	-1
18	-1	-0.5	-1
19	-1	0.5	-1
20	-1	1	-0.5
21	1	1	-0.5
22	0	1	-0.5

Fabric
-1 = #18 (OCF-117)
0 = #19 (OCF-118)
1 = #2 (OCF-107)

Temp
-1 = 550°F
0 = 650°F
1 = 750°F

A/C
-1 = 2 ft/min
0 = 4 ft/min
1 = 6 ft/min

block of tests, preformed in duplicate, will be followed by a statistical evaluation and will result in the determination of a response curve. The three fabric types selected for this series of experiments include OCF-107 (Fabric #2), OCF-118 (Fabric #19, original V/Ti catalyst on felt), and OCF-117 (Fabric #18, iron-based catalyst).

Fabric-screening tests will continue as OCF prepares new fabric samples for evaluation and in support of Subtask 3.4, Fabric Durability Testing/Pulse-Jet System, when it begins. During the next quarter, an experimental plan will be developed for the bench-scale reactor system to begin looking at the issue of catalyst poisoning.

3.3 Subtask 3.2, Process Testing/Reverse-Gas System

The project work plan states that four coals will be selected for use: two eastern bituminous coals, a western subbituminous coal, and a lignite. The four coals that EERC personnel propose to use during the pilot-scale experimental activity are listed in Tables 1 and 11, along with proximate, ultimate, and coal ash analysis.

Final selection of the baseline coal for the Task 3 pilot-scale tests was made in May. The washed Illinois #6 bituminous coal used during previous bench-scale experiments was chosen as a result of discussions held with project sponsors. Roughly 55 tons of baseline coal will be needed to complete the 100- and 500-hour tests during which the baseline coal will be used. Due to coal-storage limitations, 25 tons of washed Illinois #6 coal were ordered in June and arrived the second week in July. Thirty additional tons will be ordered in the spring of 1992. A small quantity of washed Illinois #6 coal was already available for the shakedown tests performed in August.

EERC personnel propose to use Pittsburgh #8 as the second eastern bituminous coal. Pittsburgh #8 is proposed because of its higher sulfur content and the fact that it has been used extensively by EERC for other experimental programs, including pilot-scale flue gas conditioning experiments using reverse-gas fabric filters. The Pittsburgh #8 will permit a direct performance (particulate emissions and differential pressure) comparison of the catalyst-coated reverse-gas filters with conventional woven glass filters previously used in other fabric filter tests at the EERC.

The proposed western subbituminous coal is a Wyoming Jacobs Ranch. This fuel was proposed because of its low sulfur and low ash content and its previous use during bench-scale tests with catalyst-coated fabric filter material. One of three Gulf Coast lignites will be chosen: South Hallsville, listed in Table 11; Big Brown; or Monticello. These three Gulf Coast lignites are similar. South Hallsville was used during the bench-scale catalyst-coated fabric tests, Big Brown has been used previously and is currently being used in pilot-scale flue gas conditioning experiments, and Monticello has been previously used during pilot-scale flue gas conditioning experiments.

One issue these coals do not address is the effect of alkali ash (sodium or potassium). The four fuels being considered for use during pilot-scale tests have low alkali levels. EERC had intended to address the alkali issue during Subtask 3.4, Fabric Durability Testing/Pulse-Jet System, at the UND Steam Plant. The UND Steam Plant has generally fired North Dakota lignites

TABLE 11

Coal and Ash Analyses for Three Additional Fuels Proposed for Use During the Pilot-Scale Catalytic Fabric Filter Experiments

Coal Type	Pittsburgh #8 Bituminous	Jacobs Ranch Wyoming Subbituminous	South Hallsville Lignite
Proximate Analysis (%)			
Moisture	2.6	23.1	36.8
Volatile Matter	32.1	33.0	23.6
Fixed Carbon	51.8	38.5	29.8
Ash	13.5	5.5	9.6
Ultimate Analysis (%)			
Hydrogen	4.8	6.8	6.6
Carbon	67.9	52.5	39.8
Nitrogen	1.3	0.7	0.5
Sulfur	3.7	0.3	1.3
Oxygen (Diff.)	8.8	34.3	42.2
Ash	13.5	5.5	9.6
Heating Value (Btu/lb)	12,099	9129	6719
Percent as Oxides[a]			
SiO_2	47.5	25.1	40.9
Al_2O_3	20.6	16.7	14.3
Fe_2O_3	24.4	6.4	15.9
TiO_2	1.1	1.8	1.2
P_2O_5	0.1	1.4	0.5
CaO	1.6	23.6	11.6
MgO	1.2	6.0	3.4
Na_2O	ND[b]	1.7	ND
K_2O	2.5	0.4	0.8
SO_3	1.1	16.9	14.8

[a] Weight percent concentrations of oxides are normalized values.
[b] Not detected, due to low concentration.

containing moderate- to- high-sodium levels in the ash. It appears at this time that the UND Steam Plant will be burning a low-sulfur/low-alkali subbituminous coal during the 1991/1992 and 1992/1993 heating seasons. In this event, subbituminous and lignite fuel selection for the pilot-scale experimental effort will have to be reconsidered.

The EERC project manager intends to discuss and finalize the selection of the three remaining coals during a project review meeting with project sponsors to be held in December. Procurement activities would be initiated in January 1992.

At this time, the project is roughly two months behind schedule. Replacement of the failed heating elements in the reverse-gas chambers will be necessary before Subtask 3.2, Process Testing/Reverse-Gas System, pilot-scale experimental activities can begin. Replacement heaters are expected to arrive the last week of October. The first two sets of catalyst-coated reverse-gas bags arrived on October 18, with a third set expected by the end of October. Setup and initial testing of the Siemens on-line ammonia analyzer should be completed in October if the sulfur dioxide adsorbent arrives by mid-October. Therefore, EERC is planning to perform the first 100-hour test the week of November 18, 1991, and complete two or three additional 100-hour test periods by the end of December. Holidays in November and December will prevent completion of additional 100-hour tests periods. A revised project schedule will be prepared in October and submitted to project sponsors based on the current status of the project and the balance of work to be completed.

3.4 Subtask 3.4, Fabric Durability Testing/Pulse-Jet System

Subtask 3.4 was initiated in September. Activities centered around preparation of the slipstream pulse-jet baghouse at the UND Steam Plant for use in a long-term evaluation of one set of catalyst-coated bags. Specific activities included constructing a new tube sheet, ordering new wire cages, moving the baghouse inlet from the top to the bottom of the chamber, repairing the pulse-air compressor, and adding additional heat-exchanger capacity to achieve desired temperature control. These items are nearly complete, with the exception of the repairs to the pulse-air compressor. At this time, EERC personnel hope to have a set of catalyst-coated pulse-jet bags available for installation by late December. Discussions will be held with project sponsors concerning the characteristics of the bags prior to proceeding with bag installation and initiation of the long-term test.

4.0 PUBLICATIONS AND PRESENTATIONS

An invited paper was presented at the International Joint Power Generation Conference held in San Diego, California, the week of October 6-10, 1991. The paper summarized bench-scale results upon which the pilot-scale project is based and presented project plans for the pilot-scale development effort.

5.0 KEY PERSONNEL STAFFING REPORT

Table 12 presents planned versus actual workhours for key EERC and UE&C personnel involved in the Catalytic Fabric Filtration project through September 1991.

TABLE 12

Planned Versus Actual Workhours
for Key Project Personnel Through September 1991

Key Personnel	Planned Hours	Actual Hours	Percentage of Total Available Workhours Used
Greg F. Weber	805	646	80
Dennis L. Laudal	1156	261	23
Chris Robie	29	16	55

6.0 REFERENCES

1. Pirsh, E.A. U.S. Patent 4 220 633, September 2, 1980, assigned to Babcock and Wilcox Co.

2. Pirsh, E.A. U.S. Patent 4 309 386, January 5, 1982, assigned to Babcock and Wilcox Co.

3. Doyle, J.B. et al. U.S. Patent 4 793 981, December 27, 1988, assigned to Babcock and Wilcox Co.

4. Weber, G.F. "SO_x/NO_x Control, Low-Rank Coal Research under the UND/DOE Cooperative Agreement," Quarterly Technical Progress Report, University of North Dakota Energy and Environmental Research Center, Grand Forks, ND, January 1984, DOE/FE/60181-1531, Section 6.

5. Kalinowski, M.R.; Nishioka, G.M. "Method for Applying Porous Metal Oxide Coatings to Relatively Nonporous Fibrous Substrates," United States Patent No. 4 732 879, March 22, 1988.

6. Weber, G.F.; Laudal, D.L. "SO_x/NO_x Control - Catalytic Fabric Filtration for Simultaneous NO_x and Particulate Control," Final technical report for the period April 1, 1988, through June 30, 1989; Grand Forks, ND, August 1989, DOE Contract No. DE-FC21-86MC10637.

7. Weber, G.F.; Miller, S.J.; Laudal, D.L. "Flue Gas Cleanup," Final technical report for the period July 1, 1989, through June 30, 1990; Grand Forks, ND, October 1990, DOE Contract No. DE-FC21-86MC10637.

APPENDIX A

PROCEDURE FOR BENCH-SCALE FABRIC SCREENING TESTS

PROCEDURE FOR BENCH-SCALE FABRIC SCREENING TESTS

1.0 WHILE REACTOR IS COOL, PREPARE FABRIC TO BE TESTED

If the reactor already contains a fabric sample, remove the valve and inner quartz tube. With the long metal rod provided, remove the sample. Be careful not to push too hard on the quartz frit (PATIENCE).

Select the desired fabric, and cut out three swatches using a cardboard pattern the size of a quarter (22 mm diameter).

Insert each piece of fabric into the reactor one at a time using the long metal rod. Be sure that each piece of fabric fits uniformly up against the quartz frit.

Insert the quartz inner tube, and connect the valve to the reactor. If the end of the valve does not fit tightly against the inner tube, use the teflon washer provided to fill the gap. Be sure the valve fits tightly in the reactor.

Close the furnace (DO NOT DROP), and turn on.

2.0 OPERATION OF NO_x ANALYZER

Analyzer power switch is usually left **ON** during short periods (1-2 days) of idle time. This is in order to maintain a thermal converter temperature of 650°C.

2.1 Start-Up

Valve No. 6 should be placed in N_2 position (zero gas). Open N_2 gas cylinder, and adjust supply to 10-20 psig.

Set N_2 rotameter to read 2 scfh air.

Plug in vacuum pumps (2).

Turn on ozone switch located on the front panel of the analyzer.

Open compressed air cylinder, and adjust gauge pressure to 6 psig.

Reactor chamber pressure gauge should read -28 or -29 inches Hg. If not, check vacuum pump connections, and consult owners manual.

Sample pressure gauge located on the front panel of analyzer should read -5 inches Hg. If not, open up bottom analyzer panel, and adjust screw valve located next to the air pressure gauge until sample gauge reads -5 inches Hg.

Check air gauge located inside analyzer; it should read 2 psig. If it does not, check oxygen rotameter to be sure it is open, and adjust air tank.

The N_2 rotameter controls the flow to the instrument and should be adjusted according to the valve sequence or desired function.

2.2 Calibration

2.2.1 Zero

Selection switch located on the front panel of analyzer should be in **NO** position.

Set valve to N_2 position.

Set range ppm to 100 for high span gas, 10 ppm for low span gas.

Adjust background suppression until needle reads zero. *Be sure to lock down adjustment knob when finished.

2.2.2 Span gas (high end)

Set range ppm to 1000.

Turn Valve No. 6 to NO (span gas) position.

Adjust calibration knob until needle reads 10 at far right. *Be sure to lock down adjustment knob when finished.

Return Valve No. 6 to N_2 position when done.

2.2.3 Span gas (low end)

Set range ppm to 25.

Turn Valve No. 6 to NO (span gas) position.

Adjust calibration knob until needle reads 23.6. at far right. *Be sure to lock down adjustment knob when finished.

3.0 VALVES

3.1 Measuring Inlet Conditions of NO (see Figure 1)

Turn Valve No. 7 to the open position.

Set Valves 2 and 4 to the closed position, eliminating gas flow to the reactor.

Valve No. 1 should remain in the OFF position.

Position Valve No. 6 from zero gas to sample (set necessary ppm range).

Set N_2 rotameter to read 1.5 scfh. Valve No. 5 is a bleed valve leading to the vent. This valve is used to assure positive pressure in the system and to relieve excess pressure when operating at higher flow rates.

Be sure mass flow controllers are set to the desired percent scale reading.

Read NO concentration.

3.2 Measuring Inlet Conditions of NH_3 Using NO_x Analyzer

Maintain all valve positions as in Sec. 3.1.

On the analyzer, select the NO_x position. This usually will require a greater range for the reading. The difference (absolute value) in NO concentration and NO_x concentration is the estimated NH_3 concentration.

3.3 Measuring Inlet Conditions of NH_3 Using a Dragger Tube

Maintain Valves 2 and 4 in the closed position.

Open Valve No. 1 to the vent.

Close Valve No. 7, and position Valve No. 6 to zero. Check pressure gauge, and adjust Valve No. 5 if necessary. Select colorimetric tube for appropriate concentration range.

Break off tips by placing them in the little hole located on the pump. Insert tube with arrow facing pump.

Place colorimetric tube with pump in vent line. Align red arrow on pump with line, and draw back the desired volume of sample.

3.4 Measuring NO Through Reactor

Prior to sampling through reactor, valves are usually set to measure inlet conditions.

While Valve No. 7 is in the open position, Valves 2 and 4 can be opened.

Valve No. 3 should be in the NO_x position.

Valve No. 6 should be in the sample position.

Close Valve No.7 (check rotameter and pressure gauge). The N_2 rotameter should be set at 1.5 scfh. Check pressure gauge, and use Valve No. 5 to maintain a system pressure of about 5 inches of water (at low flow rates, Valve No. 5 can be completely closed, and pressure can be adjusted with N_2 rotameter).

3.5 Measuring NH_3 Through Reactor

Maintain valve positions as in Sec. 3.4.

Select the NO_x position on the analyzer.

Allow at least 5 minutes to equilibrate, and take reading. The difference between NO_x and NO is the ammonia concentration.

Allow at least 5 minutes to equilibrate, and take reading. The difference between NO_x and NO is the ammonia concentration.

3.6 Measuring NH_3 Through Reactor Using a Dragger Tube

While valves are set for measuring with NO_x analyzer, switch Valve No. 3 to vent.

Position Valve No. 6 to zero, and proceed with Dragger tube analysis as in Sec 2.3.

When returning to analyzer measurement, switch Valve No. 6 to sample before placing Valve No. 3 in NO_x analyzer position. This will diminish the pressure build.

4.0 DETERMINING THE FLOW RATES

Flow rates are based on the desired air-to-cloth ratio and corrected for the temperature inside the reactor. Mass flow controller settings will usually be predetermined for specific operating conditions. However, in a situation where a gas cylinder is changed, the calculations would need to be redone because of a possible change in the cylinder gas concentration.

A mass balance was performed and calculated using TKSolver. The mass flow controller settings are also reported.

$$\text{Total Flow} = NO(A) + NH_3(B) + O_2(C)$$

$$0.001 \times \text{Total} = \%\ NO(x) \times \text{Flow Rate NO} \qquad (NO)$$

$$0.0009 \times \text{Total} = \%\ NH_3(y) \times \text{Flow Rate } NH_3 \qquad (NH_3)$$

$$\text{Total} - x(A) - y(B) - z(C) = (1-x)A + (1-y)B + (1-z)C \qquad (N_2)$$

Calibration curves have been done on the mass flow controllers, and their respective equations are as follows:

% scale O_2 X 80 + 146	O_2/N_2
% scale NH_3 X 2.67 - 0.6	NH_3/N_2
% scale NO X 0.87 + 0.7	NO/N_2

The desired inlet concentrations are 1000 ppm nitric oxide, 900 ppm ammonia, and 4% oxygen. The balance used to make up the desired flow rate is nitrogen. If correction of flow settings is required, update the TKSolver program with the new gas concentrations. The flow settings given by the program will vary from settings determined through operation. This is due to the difficulty in calibrating the controllers. Ammonia and nitric oxide are soluble in water, and, due to their range of flow, a bubble meter is the only calibration option.

Set the mass flow controllers to the % scale given by the TKSolver program.

Set valve sequence to measure the inlet conditions on the NO_x analyzer.

Measure the inlet NO concentration (allow at least 5 minutes to equilibrate).

While measuring the inlet concentration of NO, adjust the flow control setting until the NO_x analyzer reads 1000 ppm. While correcting the NO, adjust the O_2 (only one or two tenths of a percent) to maintain the total flow rate. Total flow rate should be back-calculated using the calibration equations.

Since the NO_x analyzer is not linear at a concentration greater than 1500 ppm the idea of a difference measurement for the determination of NH_3 would conceive a larger than measured value.

In order to determine a NH_3/NO molar ratio of 0.9, a measurement of inlet conditions using a Dragger tube should be made. In getting an initial concentration of 900 ppm ammonia and a NO measurement of 1000 ppm, a trial run should be made using a fabric of known removal efficiency.

Calculations can then be made to determine a theoretical ammonia slip.

900 ppm NH_3 - (1000 ppm - NO reading ppm)

Ammonia slip can then be checked using the difference method described in Sec. 3.5 or by the Dragger tube (Sec. 3.6)

Be sure to recalculate the total flow rate and adjust the O_2 to meet the need.

This process can become a trial and error procedure.

Once the NH_3/NO molar ratio has been established, it will be used for all other conditions in determining flow settings.

When computing flow settings for other flow rates and operating conditions, use the ratio determined in the initial setup for NO and NH_3.

$$\frac{\%\ \text{scale NO initial setup}}{\%\ \text{scale NH}_3\ \text{initial setup}} = \frac{\%\ \text{scale corrected NO}}{X}$$

Be sure to check the total flow rate and record flow settings. These settings should be used for every fabric tested at similar operating conditions.

END

DATE
FILMED
2 1 2019 2

I