**ELECTRIC POWER RESEARCH INSTITUTE
ENVIRONMENTAL CONTROL TECHNOLOGY CENTER**

REPORT TO THE STEERING COMMITTEE

Monthly Technical Progress Report

**US/DOE Patent Clearance is not Required
Prior to the Publication of this Document**

March 1997

Prepared by:

**EPRI/ECTC
Radian International
Parsons Power**

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**EPRI ECTC
7725 Lake Road
Barker, New York 14012**

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For further information on:

Facility Operations and Miscellaneous ECTC inquiries;
contact - Gary Andes (716) 795-3397

Wet FGD/Toxics Testing, Spray Drying/Dry FGD/Duct Injection technologies;
contact - Richard Rhudy (415) 855-2421

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EXECUTIVE SUMMARY

General Operations

Operations and maintenance continued this month at the Electric Power Research Institute's (EPRI's) Environmental Control Technology Center (ECTC). Testing for the month continued with the DOE/PRDA Phase I investigation of the Clear Liquor Scrubbing Process with Anhydrite Production.

The DOE/PRDA Phase I testing of the B&W/Condensing Heat Exchanger (CHX) was completed this month. This one-year tube wear analysis investigation was completed on 3/10/97, and a final inspection of the unit was made on 3/21/97. The CHX unit and its related equipment are currently being removed from the ECTC test configuration, disassembled, and returned to B&W and CHX Corp. for additional analyses.

The 1.0 MW Cold-Side Selective Catalytic Reduction (SCR) unit and the Carbon Injection System (the Pulse-Jet Fabric Filter) remained idle this month in a cold-standby mode and were inspected regularly.

Pilot Testing Highlights

In March 1997, the Clear Liquor Scrubbing with Anhydrite Production test block continued. This PRDA project is being jointly funded by the Electric Power Research Institute (EPRI) and the Department of Energy and is part of the DOE's Advanced Power Systems Program, whose mission is to accelerate the commercialization of affordable, high-efficiency, low-emission, coal-fueled electric generating technologies. The pilot portion of the CLS/Anhydrite project is being conducted on the 4.0 MW wet FGD pilot unit at the EPRI's Environmental Control Technology Center (ECTC). The project is designed to develop an advanced FGD process incorporating chloride control, clear liquor scrubbing, and anhydrite (anhydrous calcium sulfate) production.

While the three areas of the integrated process can be used independently, they work together in a manner that benefits the overall process. The HCl control process involves controlled humidification of the flue gas with a slurry containing a calcium-based alkali. The potential advantages of the HCl control process include:

- Helps control scaling in CLS process and increases the anhydrite production rate.
- Reduces the chloride concentration in FGD solutions which reduces the corrosion rate of alloys and increases the limestone dissolution rate (through calcium common ion effect).

The clear liquor scrubbing process uses an organic buffer to provide sufficient liquid-phase alkalinity to achieve the necessary SO₂ removal efficiency. The calcium sulfite solids produced from neutralizing the spent scrubber liquor with limestone are separated from the liquid in the sludge bed reactor. The resulting clear liquor

stream is recirculated to the absorber. The clear liquor scrubbing process provides the following potential benefits:

- Smaller absorber (use of enhanced mass transfer media such as packing).
- Lower L/G.
- Reduced absorber pluggage (no solids).
- Better solids dewatering properties (no recirculation).

The calcium sulfite slurry from the sludge bed reactor is sent to the anhydrite reactor for conversion to anhydrous calcium sulfate (anhydrite). The anhydrite process offers the following potential advantages:

- Byproduct differs from gypsum and is potentially more valuable:
 - “Reactive” without calcination
 - Used in other markets such as self-leveling floors, aggregate, Portland cement.
- Attractive option for either new or existing inhibited oxidation systems by producing a useful byproduct.

The original objective in the PRDA project was to demonstrate all three areas of the integrated process. However, the primary focus has been on anhydrite production for the following reasons:

- Of the three proposed processes, the anhydrite process is the least developed and potentially the most valuable,
- Initial anhydrite tests were encouraging, and
- DOE has placed emphasis on the reuse of byproduct materials.

The objectives of ECTC anhydrite test block are to:

- Produce anhydrite solids from FGD calcium sulfite slurry;
- Evaluate the effect of residence time, ionic strength, and temperature on anhydrite production;
- Characterize the anhydrite properties;
- Estimate the potential value of the anhydrite byproduct; and
- Estimate the overall production costs.

FACILITY STATUS

General

The NYSEG Kintigh Station provided flue gas to the Center 100% of the time during this performance period. As the Kintigh Station operated with a variety of coals, fluctuations in the Center's inlet SO₂ concentrations were experienced. SO₂ spiking was initiated across the test unit this month as required.

Safety training for the month was conducted by the Operations Manager, Maintenance Supervisor, and Shift Supervisors. The annual OSHA safety compliance training for the ECTC staff is currently ongoing at the NYSEG Kintigh Station. The annual ECTC employee physicals were completed at the end of the month. Inspections of the ECTC Facility and safety equipment (SCR air-packs, fire extinguishers, etc.) were completed and recorded this month. All systems were found to be in good condition. By continuing to emphasize safe work habits at the Center, we have raised the total number of days without a lost time injury to 1761 as of 3/31/97.

The monthly safety meeting with the NYSEG Kintigh Station and the EPRI ECTC was held on 3/11/97 with both NYSEG and ECTC representatives. The major topics of discussion were the completion of concurrent OSHA training and employee physicals. In addition, a request was made to NYSEG to put the ECTC on their routing list for the monthly NYSEG Safety Bulletin.

Pilot Wet FGD System

The equivalent and actual availabilities for the ECTC 4.0 MW Pilot Wet FGD System testing this month were both 99.87% during this period. The Pilot System was off-line for 1 hour on 3/3/97 due to necessary repairs across the WDPF System, DPU-5.

The DOE/PRDA Phase I Clear Liquor Scrubbing with Anhydrite Production test block continued this month with test ANH-10, ANH-11, ANH-12, ANH-13, ANH-14, ANH-15, ANH-16, and ANH-17. These tests were designed to develop the production process for the anhydrous calcium sulfate (anhydrite), and to study the solids properties of the resulting byproduct. The operational parameters for the Pilot Wet Scrubber during this test block included: gas flow of 8,250 scfm; inlet SO₂ concentrations of 2000 ppm; recycle slurry flows of 1300 to 1700 gpm; and 30 to 60 scfm oxidation air. During this performance period, a set of EPA Reference method gas samples (Method 5 and Method 2) were collected to verify the inlet particulate mass loading and flue gas flow velocity through the Pilot System.

The following maintenance work was completed on the Pilot System during this performance period:

- The stator and rotor were replaced on pump 8.
- The bearings were replaced in pump 14.
- The seal was replaced in recycle pump 9a.

- The VME was removed, cleaned and placed back in service.
- The pH probe was replaced on the MRT.
- The brushes were replaced in the centrifuge.
- A new quench spray header was fabricated and installed.
- Hoses and cam-locks were replaced on the slurry heating system.
- An O₂ Sensor was installed to monitor O₂:SO₂ Ratio on the FLRT.

Semi-Continuous Mercury Analyzer

Testing of the EPRI Semi-Continuous Mercury Analyzer was completed this month as additional comparative sampling was conducted using the analyzer and Method 29T for mercury speciation measurement. During this performance period, the analyzer and its components were moved to the particulate-free Clean Raw Gas (CRG) duct. Elemental mercury was injected (spiked) into the flue gas (prior to the ESP booster fan for good mixing) at a location upstream of the CRG sampling point for this testing. Simultaneous sampling was then conducted for total, elemental, and oxidized mercury species in the CRG duct utilizing the Semi-Continuous Mercury Analyzer and the manual sampling method (Method 29T) for comparison.

B&W/Condensing Heat Exchanger (CHX) Project

The equivalent and actual availabilities for the B&W/Condensing Heat Exchanger (CHX) unit were both 99.73% during this period. The CHX unit was off-line for 2 hours on 3/3/97 due to necessary repairs across the WDPF System, DPU-5.

Operations and testing across the B&W/CHX unit in the DOE/PRDA Phase I program were completed this month. During the performance period, a set of EPA Reference method gas samples (Method 5 and Method 2) were collected to verify the inlet particulate mass loading and flue gas flow velocity through the CHX unit.

The CHX unit was taken out of service on 3/10/97 after a final wash cycle was initiated across the heat exchanger. Samples of the wash waters were collected at discrete intervals during the wash cycle and were sent to B&W for analysis to determine the effectiveness of the new wash header design (see 2/97 report). A representative from B&W was on-site 3/21/97 to conduct a final inspection of the CHX unit and to take measurements of tube thickness in the unit. On 3/24/97, efforts began to remove the CHX unit from the ECTC test configuration and to disassemble the associated components for shipment and return to the respective project participants.

Miscellaneous

Ball Mill operation at the ECTC continued this month to provide process reagent for the Clear Liquor Scrubbing with Anhydrite Production test block. The Beachville limestone prepared for this testing was maintained at a grind of 88% passing 325

mesh screen.

The following general maintenance work was completed at the ECTC during this performance period:

- The Facility outlet CEM sample probe box was rebuilt.
- The motor for the mini-hammermill unit was replaced.
- The ESP Booster fan VSD was repaired.

Financial Report

The Financial Report for the four Operations and Maintenance contract tasks are summarized below for the month of March 1997, the year to date, and the contract total.

Task Definitions:

Task 100: Operations and Maintenance Labor

Task 200: Purchased Materials

Task 300: Engineering Support

Task 400: Training

Table i.
Monthly Operating Costs by Task

	March	Year to Date	Contract Total
Task 100	\$ -----	\$ -----	\$ 14,479,009
Task 200	\$ 2,459	\$ 8,331	\$ 8,519,527
Task 300	\$ -----	\$ 5,775	\$ 1,200,362
Task 400	\$ 5,605	\$ 5,605	\$ 58,840
DOE Grant	\$ 33,976	\$ 99,736	\$ 215,912
TOTAL	\$ 42,040	\$ 119,447	\$ 24,473,650

The attached graph illustrates the budgeted and actual expenses for ECTC O&M activities through March 1997.

Figure i.
1997 Operation & Maintenance Costs

PILOT AND MINI-PILOT TEST ACTIVITY

Testing Overview

In March 1997, the Clear Liquor Scrubbing with Anhydrite Production test block continued. This PRDA project is being jointly funded by the Electric Power Research Institute (EPRI) and the Department of Energy and is part of the DOE's Advanced Power Systems Program, whose mission is to accelerate the commercialization of affordable, high-efficiency, low-emission, coal-fueled electric generating technologies. The pilot portion of the CLS/Anhydrite project is being conducted on the 4.0 MW wet FGD pilot unit at the EPRI's Environmental Control Technology Center (ECTC). The project is designed to develop an advanced FGD process incorporating chloride control, clear liquor scrubbing, and anhydrite (anhydrous calcium sulfate) production. A conceptual process flow diagram including all three aspects of the process is included as Figure 1.

Figure 1. Conceptual Process Flow Diagram for the Clear Liquor Scrubbing with Anhydrite Production Process

While the three areas of the integrated process can be used independently, they work together in a manner that benefits the overall process. The HCl control process involves controlled humidification of the flue gas with a slurry containing a calcium-based alkali. The potential advantages of the HCl control process include:

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- Produce anhydrite solids from FGD calcium sulfite slurry;
- Evaluate the effect of residence time, ionic strength, and temperature on anhydrite production;

- Characterize the anhydrite properties;
- Estimate the potential value of the anhydrite byproduct; and
- Estimate the overall production costs.

Operating Summary

The pilot activities began following a laboratory investigation of anhydrite equilibrium and kinetics and a preliminary anhydrite market survey. The initial pilot tests focused on production of anhydrite since this is the least well developed of the three areas included in this process. The ECTC pilot configuration used in this test block is shown in Figure 2.

Figure 2. ECTC Flow Configuration Used During Anhydrite Process Development

A summary of the March operating conditions is presented in Table 1.

Table 1. March 1997 Anhydrite Operations Summary

Date	Test ID- Description	Operating Summary
Sat, 3/1	ANH-10: Same as ANH-8 with no Na addition.	Target temperature was re-established. Continued surface area control through Mechanism 2.
Sun, 3/2	ANH-10	The third absorber header was shutdown. The centrifuge product looked wetter and was extremely hard after entering the dumpster. Solids were difficult to break.
Mon, 3/3	ANH-10	Centrifuge product was different than before. Apparent increase in gypsum for unknown reason. Despite no more soda ash addition, Na conc. remains high.
Tue, 3/4	ANH-10	Took final samples. Began crushing some of the centrifuge product. The pieces first had to be broken with a hammer. The solids were not completely dry and caked the mill. The powder being ground contained about 13% hydrate.
Wed, 3/5	ANH-10	Centrifuge product dried in air was hard within about 30 minutes. Some product was also collected in isopropanol for the first time. Alcohol allows solids to dry before they react with water in the cake. Dried solids were mixed with water and set up within about one hour.
Thu, 3/6	ANH-10	Continued operation with no Na addition and Mechanism 2 for surface area. Solids washed with acetone and hot water on the filter resulted in the same % hydrate. Inlet SO ₂ was increased and third header started to build solids for lower-cost operation.
Fri, 3/7	ANH-10	Na concentration dropped slightly. Soda ash addition started again over the weekend. Centrifuge solids were collected in alcohol. The solids dumpster empties as one large rock. Operated at "weekend" conditions to build solids.
Mon, 3/10	ANH-11: Same as ANH-10 but FGD operated only weekends.	The scrubber was taken off-line in the morning. The limestone was accidentally left on and the pH increased to 5.9 in the FGDRT. The centrifuge product appeared drier but was not.
Tue, 3/11	ANH-11	Due to equipment and materials limitations, surface area control through Mechanism 2 had to be suspended. The process operated well.
Wed, 3/12	ANH-11	The liquid sulfite is significantly lower in the ART. Amount of anhydrite in product solids began to decrease. Began anhydrite surface area control through Mechanism 3.
Thu, 3/13	ANH-11	Continued Mechanism 3 for anhydrite surface area control. All variables within the target range.
Fri, 3/14	ANH-11	The scrubber was brought on line to build solids. Some sulfuric acid had to be added to the ART to briefly maintain oxidation reaction. Material limitations necessitated that anhydrite surface area control be modified to Mechanism 4.

Table 1. March 1997 Anhydrite Operations Summary

Date	Test ID- Description	Operating Summary
Sat, 3/15	ANH-11	Began adding 50# soda ash every 2 hours to maintain removal efficiency. An equipment failure forced surface area control to shift to Mechanism 5.
Sun, 3/16	ANH-11	Removal efficiency had decreased so the FGDRT pH was increased to 5.6. Limitation of materials and equipment forced a shift back to Mechanism 4 for surface area control.
Mon, 3/17	ANH-13: Same as ANH-11 but w/o surface area control.	Solids have returned to previous state of being "reactive." Not sure if this is related to change in surface area control mechanism, change in FGD operation, or unknown factor. Equipment and materials limitations have forced suspension of anhydrite surface area control.
Tue, 3/18	ANH-13	No surface area control used.
Wed, 3/19	ANH-13	No surface area control used.
Thu, 3/20	ANH-13	The centrifuge product appearance changed from wet-looking, dripping solids to drier, sandy solids. Apparently more gypsum is being produced than previously.
Fri, 3/21	ANH-13	Started the scrubber. Due to reagent addition problems, pH increased to 6.1. Centrifuge product still looks dry. Added 100# sodium thiosulfate to FGDRT. Shortly after this addition, lost the ART oxidation reaction so sulfuric acid was added. Re-established surface area control with Mechanism 4.
Sat, 3/22	ANH-14: Same as ANH-13 with Mech 4 used for surface area control	Continued Mechanism 4 in an attempt to control anhydrite surface area. pH in FGDRT maintained at 5.65 to improve removal. All other variables remained in target range.
Mon, 3/24	ANH-14	Continued Mechanism 4 for anhydrite surface area control, but the process still produced significant quantities of gypsum.
Tue, 3/25	ANH-14	Continued Mechanism 4 for anhydrite surface area control. Began preparations for an alternative method of surface area control.
Wed, 3/26	ANH-14	Mechanism 4 continued.
Thu, 3/27	ANH-14	Anhydrite surface area control Mechanism 4 continued until 19:00 hours. At that time Mechanism 6 was established for anhydrite surface area control. The centrifuge product still looked very dry. It hardened some but was easily broken apart.
Fri, 3/28	ANH-15: same as ANH-14 but with Mechanism 6 used for surface area control	Mechanism 6 used for anhydrite surface area control. Some trouble maintaining pH after starting scrubber so sulfuric acid was added as necessary.
Sat, 3/29	ANH-15	Mechanism 6 for surface area control. Continued to add sulfuric acid with air below 60 scfm.

Table 1. March 1997 Anhydrite Operations Summary

Date	Test ID- Description	Operating Summary
Sun, 3/30	ANH-15	Mechanism 6 for surface area control continued. Lost a heating coil at 7:30. When restarted at 15:00, the ART temperature was at 5°F below the target range. It increased to 1°F below the target range by midnight.
Mon, 3/31	ANH-15	ART temperature was about 2°F below the target range during the morning but dropped to 4°F below while repairing a fitting. Lowered oxidation air to 40 scfm to increase temperature. Surface area control Mechanism 6 continued.

Results Summary

Several analytical methods have been used to determine the phase of calcium sulfate being produced in the anhydrite reactor including:

- % Bound Water (measure solid weight loss at about 180°C)—provides an estimate of anhydrite wt% assuming solids are mixture of only anhydrite and gypsum;
- X-Ray Diffraction (XRD) - indicates the presence of gypsum, hemihydrate, and/or anhydrite;
- Visual Microscope and Scanning Electron Microscope (SEM)—indicate changes in particle size and shape; and
- ORTECH measurements—differential scanning calorimetry, gravimetric hydration phase analysis, and temperature setting characteristics.

Of these analyses, the % bound water and visual microscope measurements were made on site. These measurements, coupled with the subjective observations about the reactivity of the centrifuge or vacuum belt product, were used to make the daily decisions concerning modification of test conditions.

Figure 3 summarizes the solid-phase anhydrite content in the byproduct solids produced during March. The results in Figure 3 are based on the % bound water and the assumption that all of the solids are either anhydrite or gypsum. The figure shows that the anhydrite content remained above 80% for the first 2 weeks in March. The second mechanism for controlling anhydrite surface area was used from February 19 through March 11. As discussed in the February report, the anhydrite content increased to above 90% two days after surface area control mechanism 2 was begun. On March 2 (about 10 days following implementation of mechanism 2), operators made the observation that the centrifuge solids looked wetter and became extremely hard shortly after entering the dumpster used for collection. Upon cooling, the anhydrite was rehydrating with the residual water in the cake which produced an extremely hard solid. The XRD showed this material produced by the process to be mainly anhydrite with some gypsum present. SEMs of solids produced on February 8, March 5, and March 14 are shown in Figures 4, 5, and 6, respectively. The SEMs show that the predominant solid crystal shape had

changed from rods on February 8 to a “stacked platelet” shape by March 5.

On March 11, equipment and materials limitations forced a change in the surface area control mechanism. A number of different surface area control approaches were tried by the end of March. The only approach that may have been as successful as Mechanism 2 was Mechanism 5 which was used on March 15. However, using Mechanism 5 was not feasible for extended operations at the ECTC.

Following the change in surface area control mechanisms on March 11, the anhydrite content of the solids began to decrease as shown in Figure 3. By March 14, the solids were 60 to 70% anhydrite with the remainder gypsum (as determined by XRD). The SEM of the solids on March 14 indicates the presence of large gypsum crystals in addition to the smaller anhydrite crystals. The anhydrite content increased to above 80% following the use of surface area control Mechanism 5 on March 15. However, the anhydrite content decreased through the remainder of the month as other surface control approaches were implemented.

Figure 3. March 1997 Anhydrite Results Summary

Figure 4. SEM of Solids Produced at the ECTC on February 8, 1997

Figure 5. SEM of Solids Produced at the ECTC on March 5, 1997

Figure 6. SEM of Solids Produced at the ECTC on March 14, 1997

Future Plans

Further development work with the anhydrite process is planned for April. Additional experiments with surface area control will be conducted.

APPENDIX A
TEST SCHEDULES

1 9 9 5											
JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC
PILOT	High-Velocity FGD Testing	Lime/DBA Gypsum tests	Carbon Injection	Wet FGD Toxics Testing			Wet FGD Toxics Testing - Wet FGD	Outage	High-Velocity FGD Testing	Wet FGD Toxics Testing	
				Lime/DBA Gypsum tests							
MINI-PILOT	UNIT DOWN					Lime/DBA Gypsum tests	UNIT DOWN				

1 9 9 6																							
JAN		FEB		MAR		APR		MAY		JUN		JUL		AUG		SEP		OCT		NOV		DEC	
PILOT	EPRI Wet FGD Toxics Testing			DOE/PRDA Phase I - Mercury and HAPs Control with Wet FGD				EPRI Wet FGD Toxics Testing				Third party Testing - Rotorfilter Technology		EPRI Toxics Removal Testing		EPRI SOx/NOx Process Testing		ECTC Fall Outage					
	UNIT DOWN			DOE/PRDA Phase I - Multiple Pollutant Removal Using the Condensing Heat Exchanger (CHX)																			
MINI-PILOT																							

1 9 9 7 (Tentative Schedule)											
JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC
DOE/PRDA Phase I - Clear Liquor Scrubbing (CLS) with Anhydrite Production				EPRI, ECTC, and Third-Party Environmental Testing (API Phase II)					DOE/PRDA Phase II - Mercury and HAPs Control		DOE/PRDA Phase II - Clear Liquor Scrubbing
DOE/PRDA Phase I - Condensing Heat Exchanger (CHX) Testing			Reconfigure Mini-Pilot Scrubber	Unit Available for Testing							

ECTC Pilot-scale Testing Schedule ('95-'97)

ECTC COSPONSORS DISTRIBUTION

Mr. John E. Smigelski
New York State Electric & Gas
Corporate Drive, Kirkwood Ind. Park
P. O. Box 5224
Binghamton, NY 13902-5224
Ph: (607) 762-8688
Fax: (607) 762-8457

Ms. JoAnn C. Zysk
U.S. Department of Energy
Federal Energy Technology Center
P. O. Box 10940, MS 900-33
Pittsburgh, PA 15236
Ph: (412) 892-6200
Fax: (412) 892-6216

Dr. Gerald Janik
New York State Electric & Gas
Corporate Drive, Kirkwood Ind. Park
P. O. Box 5224
Binghamton, NY 13902-5224
Ph: (607) 762-4318
Fax: (607) 762-8457

Mr. Takeo Hirai
Electric Power Development Co., Ltd.
15-1, Ginza 6-Chome, Chuo-ku
Tokyo, 104 JAPAN
Ph: (81) 3-3546-9404
Fax: (81) 3-3546-9482

Ms. Debra DiMeo
Empire State Electric Energy Res. Corp.
1515 Broadway
New York, NY 10036
Ph. (212) 302-1212
Fax: (212) 302-1377

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