

Commonwealth Energy Biogas/PV Mini-Grid
Renewable Resources Program

***Making Renewables Part of an Affordable and
Diverse Electric System in California***

Contract No. 500-00-036

Fifth Quarterly Data Report

Data for the Period June 1 – September 30, 2005

Project 2.2 Enhanced Energy Recovery through Optimization
of Anaerobic Digestion and Microturbines

Task 2.2.5 Collect and Analyze Data for Optimized Anaerobic Digestion System

Prepared For:
California Energy Commission
Public Interest Energy Research Renewables Program

Prepared By:

CH2MHILL

Bill Kitto, CH2M HILL, Santa Ana, California
3 Hutton Centre Drive, Suite 200
Santa Ana, CA 92707

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Acronyms and Abbreviations

AFT	Applied Filter Technology
AMD	acid manure digester
BFP	belt filter press
Btu/cf	British thermal units per cubic foot
cf/d	cubic feet per day
cf/hr	cubic feet per hour
cf/lb	cubic feet per pound
cfm	cubic feet per minute
CO ₂	carbon dioxide
DAF	dissolved air flotation
DAFT	dissolved air flotation thickener
dtpd	dry ton per day
FID	flame ionization detector
FS	fixed solids
g/cm ³	gram per centimeter cubed
gpm	gallon per minute
H ₂ S	hydrogen sulfide
HRT	hydraulic retention time
IA/PA	volatile acids (VA): Alkalinity ratio (expressed as "IA/PA" in IEUA analytical protocols)
IEUA	Inland Empire Utilities Agency
K cf/d	1,000 cubic feet per day
K cf/hr	1,000 cubic feet per hour
K gal/d	1,000 gallons per day
K gpd	1,000 gallons per day
K lb/d	1,000 pounds per day
lb/d	pound per day
lb/gal	pound per gallon
µg/L	micrograms per liter
MG	million gallon
mgd	million gallon per day
mg/L	milligram per liter

NRW	non-reclaimable waste
PIER	Public Interest Energy Research
ppm	part per million
ppmv	part per million by volume, dry
PS	primary sludge
psig	pound per square inch gauge
SiO ₂	silicone dioxide
SLR	solids loading rate
SWD	side water depth
TDS	total dissolved solid
TKN	total Kjeldahl nitrogen
TPS	thickened primary sludge
TS	total solids
TSS	total suspended solid
TWAS	thickened waste activated sludge
VA	volatile acids
VS	volatile solids
VSR	volatile solids reduction
WAS	waste activated sludge
wtpd	wet ton per day

Executive Summary

Background

The *Process Selection Report for Wastewater Treatment Plants*, delivered under Task 2.2.1 (Project 2.2) of the Public Interest Energy Research Renewables (PIER) program, evaluated several different processes, and recommended focusing on ultrasound testing for enhanced anaerobic digestion and a custom treatment package for microturbine biogas cleaning.

The *Site Selection and Test Plan Report*, delivered under Task 2.2.2 (Project 2.2) of the PIER program, carried forward the conclusions from the above-mentioned reports and provided specific site recommendations, further definition of the processes and their integration into the host facility at the recommended site, and the test plan for the recommended processes.

Ultrasound Testing

The ultrasound process selected in Task 2.2.1 was recommended for testing at the City of Riverside Water Quality Control Plant. A specific location on the south side of digesters #1 and #2 at that plant was recommended and described on a plant layout. Two vendors of ultrasound systems, IWE Tec and Sonico, were selected to provide equipment. The recommended test plan was broken down into four phases: a pretest phase (preparing for the test), a baseline phase (with no ultrasound system installation), an ultrasound phase (with ultrasound system installation), and a continuation phase (after the shutdown of the ultrasound systems).

Microturbine Gas Cleaning

The microturbine gas cleaning test was recommended for testing at the Inland Empire Utilities Agency's Regional Plant 1 (IEUA RP-1). The recommended microturbine gas cleaning pilot system consists of a refrigerated dryer system for moisture removal, a packaged system for siloxane removal in the biogas and a biological hydrogen sulfide (H₂S) removal scrubber. The recommended test plan was broken down into three phases: a pretest phase, a baseline phase, and an actual testing phase. During the pretest phase, the size of the different equipment for the pilot test program was optimized. The baseline for H₂S removal in Digester No. 4 is the existing method of ferric chloride injection. Once the equipment is installed, the biological H₂S removal testing phase will start. For the gas drying and packaged siloxane removal system, the baseline testing was conducted during two sample rounds in October and November 2004 and continuing weekly siloxane sampling on the combined gas. A continuation test phase is not required for the microturbine gas cleaning pilot program.

Quarterly Reports

The first quarterly data report submitted in December 2004 summarized the baseline test results obtained from June 1 to August 31, 2004, for the enhanced anaerobic digestion using ultrasound. The second and third quarterly reports presented the test results of the

ultrasound test phase for the periods from September 1 to November 30, 2004, and December 1, 2004, through February 28, 2005, respectively. In addition, the baseline data were included for the microturbine gas cleaning test at RP-1. The ultrasound performance provided site-specific digester performance for gas production, biosolids production, and various other parameters based on the known amount of primary sludge (PS) and thickened waste-activated sludge (TWAS) fed to digesters. The data were used for comparing digester improvements with the ultrasound systems installed. The gas cleaning test baseline data mainly included baseline H₂S level in the biogas with ferric chloride addition, H₂S removal by the existing iron sponge system, and moisture and siloxane levels in digester gas. The data from the baseline test phase will be used for comparing moisture, H₂S, and siloxane removal by each recommended pilot testing system.

The fourth quarterly report presented the test results of the continuation phase of the ultrasound testing project that covered the period from March 1 to May 31, 2005. The continuation phase was conducted to continue monitoring digester performance after the shut down of the ultrasound equipment. Since the 1-year enhanced anaerobic digestion ultrasound testing at Riverside Plant was completed by the end of the continuation phase, this quarterly report also summarized the findings from the complete test. The other component in this fourth quarterly report was the microturbine gas cleaning project at RP-1. Additional baseline siloxane data obtained during the same time period of March to May 2005 were included in this report.

This fifth quarterly report presents continuing baseline data for siloxane concentration in the combined gas and Digester 1 and 2 gas streams at RP-1. Digester 4 H₂S production and control with ferric chloride baseline data is also presented for comparison with performance of the biological scrubber during the test phase.

Microturbine Gas Cleaning—Baseline Period

To establish baseline performance, the operation and performance of the digesters and gas treatment systems at RP-1 were monitored for a number of key performance parameters, such as ferric chloride addition, feed flow and total solids (TS) fed to the digester, digester gas production, H₂S concentration in the digester gas, and siloxanes in the combined gas before the iron sponge system.

In addition to the regular digester monitoring, two rounds of gas sampling were conducted to determine the reduced sulfur compounds, moisture content, siloxane, and volatile organic compounds (VOCs), as outlined in the pretest plan. The samples were taken on October 12 and November 16, 2004, and the results were presented in the 2nd quarterly report.

Baseline for Digester Gas Siloxane Levels

Siloxanes are chemicals used extensively in industrial, personal care, and food products. They can therefore enter wastewater from a number of different sources, usually in a stable emulsion form. However, under digester conditions, they volatilize into the digester gas and form silicone dioxide (SiO₂) deposits during combustion that can cause severe damage in generator engines, boilers, and microturbines. Siloxanes were tested in the samples taken

from the two rounds collected in October and November of 2004. Additional samples have been collected by plant staff since July 2004. Gas samples were taken from individual digesters and the combined gas line in various locations, such as combined gas at the flare, before and after the iron sponge system, and after the gas compressor. Two methods were used for measuring the siloxanes content of digester gas – the canister method and the impinger method. Eight selected siloxane compounds most commonly found in biogas were analyzed and the total siloxane calculated.

Over the entire baseline period the siloxane results for the combined gas have varied from 3 to 4,243 micrograms per liter ($\mu\text{g}/\text{L}$). The siloxane levels are usually significantly higher than the levels for microturbine operation requirement. For comparison, siloxanes were also analyzed in biogas samples collected from the Riverside plant digesters on November 16, 2004, and showed levels less than 5 ppmv.

The siloxane levels ranged from 25 to 3,124 $\mu\text{g}/\text{L}$ for digesters 2, 3, 6, and 7 from two rounds of tests using canister method and additional impinger sampling conducted on Digester 2. Digester 1, the mesophilic acid digester, has had siloxane levels ranging from 2 to 1,357 $\mu\text{g}/\text{L}$ using both Impinger and Canister methods. Digester 4, the manure digester, had non-detectable siloxanes from tests performed in July and October 2004

Baseline for H₂S Removal

The following methods of gas treatment are currently used:

- Ferric chloride addition to headworks for sludge digester gas H₂S control
- Ferric chloride addition directly to manure Digester 4 for digester gas H₂S control
- Iron sponges for H₂S removal from the combined digester gas stream

Since the biological H₂S scrubber will be tested in the gas from Digester 4, the H₂S production from this digester was monitored to provide the baseline operation parameters. Baseline data from routine digester gas H₂S monitoring for the period of January 2005 through September 2005 showed the monthly average H₂S in the digester gas ranged from 41 to 70 ppmv (average 56 ppmv), and the ferric chloride addition averaged 134 gpd, or 1,648 pounds per day (lb/d) at 40 percent active. The ferric dose and the digester gas H₂S concentration did appear to be affected by foodwaste addition to the digester.

The cost for purchasing ferric chloride is \$318/ton (includes 7.75 percent tax), according to the plant staff. The average daily cost for ferric chloride was \$251, which corresponds to 0.8 cent per gallon of digester feed, 0.7 cent per pound of TS feed, or 0.2 cent per cubic feet biogas generated; and \$14 per pound of H₂S removed from the biogas through addition of ferric chloride, assuming that the H₂S concentration would be 2,000 ppm without treatment.

Next Phase—Gas Cleaning Pilot Test Phase

The next phase consists of gas cleaning pilot testing. The pilot refrigerated dryer (chiller) system, siloxane removal media, and biological H₂S removal system are currently underway. The chiller and siloxane system are expected to be in operation by October 2005, as is the biological H₂S system.

Information on equipment and instrumentation needed to conduct the pilot test, test procedures, and the sampling plan (parameters and testing frequencies) are provided in Section 5 of this report.

Introduction

In June 2001, the Commonwealth Energy Team was awarded a programmatic contract under the California Energy Commission's Public Interest Energy Research (PIER) Program to conduct research on strategies for making renewable energy more affordable in California. The Commonwealth Energy approach involves assessing the combined potential of biogas and photovoltaic (PV) resources in a defined study area and identifying how these resources could be developed in a complementary and cost-effective manner. The Commonwealth Energy Team conducted this research in a real world setting so that the findings could be applied elsewhere in California and thereby benefits more California ratepayers. The local area Commonwealth Energy selected for its renewable energy research activities is the Chino Basin, referred to in this report as the study area.

1.1 Background

The Chino Basin is rich in PV and biogas resources. Moreover, it is a rapidly growing area with substantial and increasing electrical loads. The underlying goal of the Commonwealth Energy PIER Renewables Mini-Grid Program is to identify potential Building Integrated PV (BIPV) and biogas energy projects, bring innovative technologies and business practices to these projects, assess the benefit to the local electricity distribution system (the mini-grid), and then use the findings to develop a business model for siting cost-effective, renewable energy projects. A description of the Commonwealth Energy PIER Program, including the results of some of the work undertaken to date, is presented on the project Web site, <http://www.pierminigrid.org>.

An important element of the Commonwealth PIER Renewables Mini-Grid Program is a project devoted to research on improving energy recovery from biogas derived from anaerobic digestion. This project is identified as Project 2.2, "Enhanced Energy Recovery Through Optimization of Anaerobic Digestion and Microturbines." The work summarized in this report, Task 2.2.5 "Collect and Analyze Data for Optimized Anaerobic Digestion System," is the fifth activity of Project 2.2. This task requires providing quarterly data reports on data collection and analysis activities.

1.2 Overview of Project 2.2

The Project 2.2 is entitled "Enhanced Energy Recovery through Optimization of Anaerobic Digestion and Microturbines". The objectives of Project 2.2 are to:

- Increase and optimize digester gas production through thermal hydrolysis and ultrasound processes
- Develop and optimize cost-effective gas cleanup systems

- Evaluate and quantify environmental benefits that result from using microturbines at sewage treatment plants
- Evaluate performance and cost during operation so sewage treatment plants have greater certainty on cost and reliability of using microturbines

The first task, 2.2.1, evaluated several different processes and selected an ultrasound process for enhanced anaerobic digestion and a custom treatment package for gas cleaning of microturbines, to be carried further to site selection and testing.

The *Site Selection and Test Plan Report*, delivered under Task 2.2.2 (Project 2.2) of this PIER program, carried forward the conclusions from the above-mentioned report and provided specific site recommendations, further definition of the processes and integration into the host facility at the recommended site, and the test plan for the recommended processes. The second Task 2.2.2 included a report on selection of the best sites at which to deploy the technologies and processes for enhanced anaerobic digestion that were selected in Task 2.2.1. It also provided (1) expanded process flow diagrams that further define the selected processes and show integration into the selected host facility, and (2) the test plan for the new systems.

Four quarterly data reports have been prepared prior to this report. These included the baseline, test and continuation phases of the ultrasound enhanced digestion test conducted at the City of Riverside Regional Water Quality Control Plant, and baseline data for the gas cleaning test.

1.3 Aim of this Report

This report provides the continued baseline data for the microturbine gas cleaning tests at Inland Empire Utilities Agency's Regional Plant 1 (IEUA RP-1). The fourth quarterly report submitted previously provided the conclusion of the ultrasound test that was conducted at the City of Riverside Regional Water Quality Control Plant, therefore this report is focused on the gas cleaning test.

- **Microturbine Gas Cleaning Tests at IEUA's RP-1:** This report summarizes the test gas cleaning data for both sewage sludge and manure digestion, for the period June 1 through September 30, 2005. The gas cleaning test focuses on removing H₂S, moisture, and siloxane for microturbines. The baseline gas cleaning data include the regular digester operation and performance with respect to siloxanes in the digester gas and H₂S in the Digester 4 gas. Additional laboratory tests performed for reduced sulfur compounds, siloxane, moisture content, volatile organic compounds (VOCs) were conducted in October and November 2004 and have been reported in the second quarterly report.

1.4 Report Content and Organization

This report is organized as follows:

- **Section 1** introduces the Commonwealth Energy program, provides background information on the Chino Basin, presents an overview of the Commonwealth PIER

project for Enhanced Energy Recovery through Optimization of Anaerobic Digestion and Microturbines, and describes the objectives and content of this report.

- **Section 2** provides an overview of the microturbine gas cleaning project summary and evaluation of the Chiller and **SagPack™** unit performance (June 1 to September 30, 2005). Digester performance is compared.
- **Section 3** provides a summary of additional baseline data for siloxanes at the RP-1 plant (June 1 to September 30, 2005).
- **Section 4** provides a summary of additional baseline data for H₂S produced by Digester 4 at the RP-1 plant (June 1 to September 30, 2005).
- **Section 5** describes the next steps for starting the pilot test phase in the next quarter.
- **Section 6** describes quality assurance and data analysis procedures followed for the data collected through the fifth quarter.

1.5 Task 2.2.5 Scope and Deliverables

The scope for task 2.2.5 is to submit quarterly data reports on data collection and analysis activities. Gas quantity and quality information is to be collected before and after installation.

The work statement for task 2.2.5 lists data to be collected as follows:

- The data to be collected after installation to analyze the performance of the microturbine and associated gas cleaning systems include power generation, heat recovery, air emissions, construction costs, and operating costs. The equipment's performance will be measured in terms of heat rate, reliability, and emissions. Analyses will be conducted and compared to predicted values for these data.
- The data will also be collected to analyze the effectiveness of the systems installed for anaerobic digestion gas production optimization. Information collected prior to installation on flow rates, solids quality and quantity, digester loading rates, process recycles, and gas production will be compared to similar data collected after installation. Installation, operation, and maintenance cost information will be collected. The analysis process will focus on determining the impact on process operation.

The deliverables for Task 2.2.5 are:

- a) Quarterly Data Report # 1
- b) Quarterly Data Report # 2
- c) Quarterly Data Report # 3
- d) Quarterly Data Report # 4
- e) Quarterly Data Report # 5

The microturbine and gas cleaning systems pilot equipment was installed at IEUA's RP-1 facility. The pilot equipment for anaerobic digestion gas production optimization was installed at the City of Riverside Regional Water Quality Control Plant. Due to the different

timing of these two separate installations, the test for anaerobic digestion gas production optimization was completed before the microturbine and gas cleaning systems pilot equipment was in operation. Therefore, the 5th quarterly report, for the period of June 1 to September 30, 2005, includes only continuing baseline data for microturbine and gas cleaning systems at RP-1.

Test data points for the parameters listed above for microturbine and gas cleaning systems pilot testing are included in this report as follows:

- **Power Generation** – not taken during the continuing baseline phase in the 5th quarter
- **Heat Recovery** – not taken during the continuing baseline phase in the 5th quarter
- **Air Emissions** – **Section 3** (siloxanes), **Section 4** (H₂S)
- **Construction and Operating Costs** – Installation for gas treatment systems was not complete; therefore, those costs are not available for the 5th quarterly report and will be summarized in the subsequent quarterly report. Baseline operating costs for gas treatment at RP-1 for the period are discussed in **Sections 3 and 4**

Microturbine Gas Cleaning Overview

This section provides an overview of microturbine gas cleaning test technologies, describes the gas cleaning pilot program, and presents additional test data obtained during the period of June 1 through September 30, 2005. During this phase the chiller and SagPack™ system was started up. However, due to operational issues with the microturbines, flow through the system was too low and the system was shut down to allow re-routing of the treated gas stream back into the feed to the IC engines. The treated gas data obtained during this quarter are not representative of proper operation of the chiller and SagPack™ system and are therefore not reported.

2.1 Overview of Gas Cleaning Tests

The biogas used for operating microturbines must meet stringent quality requirements (maximum of 150 parts per million [ppm] moisture, 25 ppm H₂S, and 10 parts per billion by volume [ppbv] siloxanes) to prevent early deterioration of the microturbines. The biogas produced in the digesters at a wastewater treatment plant is typically saturated and contains approximately 500 to 2,000 ppm H₂S, and 2 to 5 ppm siloxane. Biogas from manure digestion is also saturated and contains approximately 500 to 2,000 ppm H₂S with no siloxanes. The content of each of these contaminants needs to be decreased to meet the biogas quality stated above.

This section provides an overview of the gas cleaning test, including the test purpose, pilot test site location, technologies to be tested, a process flow diagram, and the test plan.

2.1.1 Gas Cleaning Test Purpose

The purpose of the microturbine gas cleaning pilot test is to collect and analyze data for different technologies to determine their efficacy in removing hydrogen sulfide, moisture, and siloxanes.

2.1.2 Gas Cleaning Test Location

The RP-1 facility has been selected to conduct the biogas cleaning pilot test program because it has microturbines and biogas is generated using both municipal waste and manure.

2.2 Existing Gas Quality Control at RP-1

RP-1 has seven anaerobic digesters (Nos. 1 through 7). The biogas system consists of an iron sponge system to remove H₂S, biogas compressors and storage, an energy recovery building, a waste gas burner, and eight microturbines. Six of the seven digesters (digesters 1 through 3 and 5 through 7) at RP-1 process the solids from sewage wastewater treatment. Digester 4 is used to process dairy manure.

To control H₂S in the biogas from digestion of municipal waste, iron salts are added at the headworks to bind the sulfur as iron sulfide. To reduce H₂S levels in the manure digester biogas, iron salt is added directly to this digester. After the biogas is collected from all the digesters, it is treated through iron sponges, further reducing the H₂S concentration from around 60 ppm to 20 ppm on average. Under normal operation, the gas from Digester 1 (the acid digester) is directed to the waste gas burner as the low methane content of the gas from Digester 1 reduces the performance of the co-generation engines. The biogas compressors are located downstream of the iron sponges and increase the biogas pressure to around 60 pounds per square inch gauge (psig) before it is stored in the biogas storage system. From the storage system, the biogas is distributed to the engine generators, boilers, and microturbines. There is no siloxane removal for the internal combustion engines, but carbon filters were installed to reduce siloxane levels in the biogas used in microturbines.

2.3 Gas Cleaning Test Technologies

The proposed gas cleaning pilot systems consist of testing technologies that have the potential to:

- Remove moisture through gas drying through a refrigerated dryer system that consists of a refrigeration unit, and two heat exchangers. The gas drying system may also bring the benefit of some siloxane removal through the condensate.
- Remove H₂S in biogas through a biological process. The bacteria in this process oxidize the sulfide to produce both elementary sulfur and sulfuric acid.
- Remove siloxanes through packaged systems. SAGPack series manufactured by Applied Filter Technology (AFT) was selected for pilot testing. A new media will be tested in parallel with AFT's standard media.

None of the test technologies have been used in the U.S. or been applied at the scale needed for microturbine gas treatment.

2.4 Gas Cleaning Test Plan at RP-1

The process flowchart for the proposed pilot test, equipment needed to conduct this test, sampling locations, parameters to be tested, and testing frequencies were discussed in detail in the Task 2.2.2 *Site Selection and Test Plan Report*.

2.4.1 Gas Cleaning Test Recommendations and Schedule

The test recommendation was to proceed with pretesting of the biogas to optimize the size of the different equipment for the pilot test program (gas drying chiller, biological H₂S removal scrubber, and Sagpack siloxane removal media) and provide baseline data and costs.

The existing method for removal of H₂S in Digester 4 (ferric chloride injection) needs to be monitored to establish the removal efficiency baseline for this H₂S removal technology. Additional baseline testing was conducted during two sample rounds in October and

November 2004 to test for reduced sulfur compounds in the digester gas, and sulfur concentrations in the digester feed and effluent.

Baseline testing for the chiller and Sagpack system was conducted during two detailed rounds of sampling on each digester in October and November 2004, and by weekly siloxane sampling of the Digester 1 and combined gas streams. During the two detailed sample rounds, gas from each digester and the combined gas were tested for siloxanes, moisture, and VOCs. For the Microturbine Gas Cleaning pilot program, a continuation test phase is not required. Table 2-1 summarizes the time requirement for the different technologies and corresponding phases.

TABLE 2-1
Microturbine Gas Cleaning Test Duration

Phase	Duration	Status
Pretest Phase (all pilot technologies)	1 month	Completed
Baseline Phase Additional Sampling	1 month	Completed
Biological H ₂ S Removal Phase	3 months	Expected start Oct. 2005
Gas Drying and Package System Phase	3 months	Re-start after modifications Oct. 2005
Iron Sponge Air Injection Test Phase	3 months	Expected start Oct. 2005

Additional Baseline Data for Siloxane Quality

The baseline data collected for both the biological H₂S removal system and moisture/siloxane removal by Chiller/Sagpack were presented in the Second Quarterly Report. Additional baseline siloxane data have been presented in subsequent reports. This section presents additional baseline siloxane test data obtained during June through September 2005. Siloxanes are chemicals used extensively in industrial, personal care and food products. They can therefore enter wastewater from a number of different sources, usually in a stable emulsion form. However, in the conditions in a digester, they volatilize into the digester gas, and form silicon dioxide (SiO₂, also known as silica) deposits during combustion that can cause quite severe damage in generator engines and microturbines.

3.1 Siloxane Sampling Methods

Two sampling methods were used for collecting the digester gas samples for siloxanes measurement, the canister and the impinger methods. There is currently no EPA-approved methodology for gas siloxane sampling and analysis. For this study, digester gas canister sampling grabs samples using Summa® canisters were collected from the digester gas lines. Sample lines were 0.25-inch Teflon, and sampling location had positive pressure (greater than 5-inch water column positive pressure). EPA Method TO-15 was used for laboratory analysis of the samples. The sampling protocol provided by an independent sampling specialist is attached in Appendix A of this report. The advantages and disadvantages of the canister method are summarized below.

- Advantages:
 - Shorter sampling time
 - Lower limits of detection
 - May be more accurate
- Disadvantages:
 - Siloxanes can be adsorbed onto the surface of stainless steel canisters if not properly pretreated
 - Siloxane standards are not readily available to laboratories
 - Short-term sample

In the impinger method, siloxanes are absorbed into liquid methanol using impingers. Samples are drawn into the impinger with a vacuum pump at a known flow rate over a period of 3 to 4 hours. Samples are analyzed by injection of the methanol into a GC/MS or GC/flame ionization detector (FID). The siloxanes concentration in the gas is back calculated using the measured gas flow rate through the methanol and the duration of sample collection. The advantages and disadvantages of the impinger method are summarized below.

- Advantages:
 - Long-term composite sample
 - Possibility of better limits of detection
 - Widespread acceptance
- Disadvantages:
 - Time-consuming in the field
 - May not capture 100 percent of siloxanes

Eight selected siloxane compounds that are most commonly found in biogas were analyzed and reported and the total siloxanes can be summed.

3.2 RP-1 Siloxane Data

Table 3-1 summarizes the siloxane results from both canister and impinger methods for individual digester and combined gas during the baseline period. Combined gas samples were taken from various locations in the gas system, such as combined gas at flare, before and after iron sponge system, and after compressor. Figure 3-1 is the graphic presentation of total siloxanes in the combined digester gas in $\mu\text{g}/\text{L}$.

During September and October 2004, the RP-1 staff noticed a buildup of white material in the Waukesha IC engines at the plant. Analysis of the material by the plant laboratory showed that it was primarily composed of silicon. The baseline siloxane data support this, as concentrations in the October gas samples were significantly higher than in a sample collected in July 2004, which had a total siloxane concentration of $18 \mu\text{g}/\text{L}$ (2 ppmv). The concentration remained high in November, however, it was lower in the following months, with occasional high spikes. The total siloxane results for the combined gas varied from 3 to $309 \mu\text{g}/\text{L}$, lower than the previous quarter. The cause of the initial prolonged increase in siloxane concentrations and the subsequent spikes has been difficult to determine as siloxanes in the influent wastewater are in a very stable emulsion form that is not amendable to laboratory analysis. Without any treatment, the siloxane levels are significantly higher than the microturbine operation requirement. Internal combustion engines have a less stringent requirement, with a maximum siloxane level of $25 \mu\text{g}/\text{L}$ (1.8 ppmv) for Waukesha engines.

Figure 3-2 presents the total siloxane results for Digester 1 using both canister and impinger methods and for Digester 2 using the impinger method. Digester 1 is the mesophilic acid-phase digester, it is operated with low HRT (less than 5 days) and low pH (around 5.3) to favor acid producing bacteria from the organics in the wastewater sludge. The produced acid is further converted to methane in the following second-phase gas digesters. Digester 2 is a gas phase digester, typically operated at low thermophilic temperatures (126°F) and a HRT around 11 days. Between June and September 2005, siloxane concentrations in the gas samples collected from Digester 1 remained in the range of 20 to $26 \mu\text{g}/\text{L}$, which is around the limit recommended for the Waukesha engines. One Digester 2 sample was collected in September and showed a similar concentration to Digester 1, at $25 \mu\text{g}/\text{L}$. Table 3-1 provides the siloxane results from all the digester gas samples taken at the RP-1 plant since July 2004. Comparison of Digester 1 and Digester 2 concentrations for samples collected on or close to the same days show that both digesters had similar siloxane concentrations.

TABLE 3-1
 Siloxane in the RP-1 Digester Gas (µg/L) (July 2004 to September 30, 2005)

Sampling Date	Sampling Location															
	Dig 1		Dig 2		Dig 3	Dig 4	Dig 6	Dig 7	Combined Gas Digs 1, 2, 3, 4, 6, 7 (Flare)		Combined Gas Digs 2, 3, 4, 6, 7 (Flare)		Combined Gas Digs 2, 3, 4, 6, 7 (Before Iron Sponge)	Combined Gas Digs 2, 3, 4, 6, 7 (After Iron Sponge)	Combined Gas Digs 2, 3, 4, 6, 7 (After Compressor, Engine)	
	Can	Imp	Can	Imp	Can	Can	Can	Can	Can	Imp	Can	Imp	Imp	Can	Can	Imp
7/6/04						ND			18							
10/12/04	1,357		880		1,077	ND	3,124	455	477							
(Dup) 10/12/04									983							
10/20/04	372								650							
10/25/04	271	62							227	72						
11/2/04	21										16					
11/3/04		47										124				
11/10/04		306											1,066			9
11/16/04	94	42	78		30		70	40					1,850	191	118	
(Dup) 11/16/04													1,740		72	
11/23/04		13											245			
(Dup) 11/23/04		12											236			
12/3/04		24														91
12/9/04		16														69
12/16/04		33														129
12/22/04		103														97
1/5/05		22														32
1/13/05		8														190
1/19/05		7														176
(Dup) 1/19/05																173
2/15/05		8														20
2/24/05		14														54
03/03/05		23												73		
03/10/05				263										763		
(Dup) 03/10/05				294												
03/15/05				89										4,243		
(Dup) 03/15/05				76												
03/18/05		80									12					
(Dup) 03/18/05		85														
03/23/05		2												16		

TABLE 3-1
Siloxane in the RP-1 Digester Gas ($\mu\text{g/L}$) (July 2004 to September 30, 2005)

Sampling Date	Sampling Location															
	Dig 1		Dig 2		Dig 3	Dig 4	Dig 6	Dig 7	Combined Gas Digs 1, 2, 3, 4, 6, 7 (Flare)		Combined Gas Digs 2, 3, 4, 6, 7 (Flare)		Combined Gas Digs 2, 3, 4, 6, 7 (Before Iron Sponge)	Combined Gas Digs 2, 3, 4, 6, 7 (After Iron Sponge)	Combined Gas Digs 2, 3, 4, 6, 7 (After Compressor, Engine)	
	Can	Imp	Can	Imp	Can	Can	Can	Can	Can	Imp	Can	Imp	Imp	Can	Can	Imp
03/31/05		14														12
04/07/05		18														172
(Dup) 04/07/05		19														
04/13/05		15														44
04/20/05		2														617
(Dup) 04/20/05																586
04/21/05																
04/27/05																
04/28/05		7														17
05/04/05		19														36
(Dup) 05/04/05		19														36
05/09/05																
05/11/05		23														855
05/20/05		8														114
6/2/2005		20											66			
6/8/2005		25											309			
7/20/2005									5							
7/21/2005		26											145			
7/26/2005																
7/28/2005									3							
8/4/2005									65							
8/10/2005									100							
9/1/2005																
9/9/2005																
9/20/2005																
9/21/2005				25									135			
(Dup) 9/21/2005													124			

Notes:

ND = not detected.

Dup: duplicate samples

Can = Canister Method, Imp = Impinger Method

TABLE 3-1
 Siloxane in the RP-1 Digester Gas (µg/L) (July 2004 to September 30, 2005)

Sampling Date	Sampling Location															
	Dig 1		Dig 2		Dig 3	Dig 4	Dig 6	Dig 7	Combined Gas Digs 1, 2, 3, 4, 6, 7 (Flare)		Combined Gas Digs 2, 3, 4, 6, 7 (Flare)		Combined Gas Digs 2, 3, 4, 6, 7 (Before Iron Sponge)	Combined Gas Digs 2, 3, 4, 6, 7 (After Iron Sponge)	Combined Gas Digs 2, 3, 4, 6, 7 (After Compressor, Engine)	
	Can	Imp	Can	Imp	Can	Can	Can	Can	Can	Imp	Can	Imp	Imp	Can	Can	Imp

To convert µg/L to ppmv, use: ppmv = µg/L *23.68/333.69/1,000

3.3 Summary of Baseline Digester Gas Siloxane Levels

Gas samples were taken from individual digesters as well as combined gas lines in various locations such as combined gas at the flare, before and after the iron sponge system, and after the gas compressor. Two methods were used for measuring the siloxanes content of digester gas – the canister method and the impinger method. Eight selected siloxane compounds most commonly found in biogas were analyzed and the total siloxane calculated. The siloxane results obtained from July 2004 through September 2005 (baseline phase) indicate that:

- Siloxane levels in the combined gas varied significantly from 3 to 4,243 $\mu\text{g}/\text{L}$ over the entire period. Siloxane levels were often significantly higher than operation requirements for microturbine and generator engines and therefore the gas requires treatment.
- The siloxane levels ranged from 25 to 3,124 $\mu\text{g}/\text{L}$ for digesters 2, 3, 6, and 7 from two rounds of tests using canister method and additional impinger sampling conducted on Digester 2. Digester 1, the mesophilic acid digester, has had siloxane levels ranging from 2 to 1,357 $\mu\text{g}/\text{L}$ using both Impinger and Canister methods. Digester 4, the manure digester, had non-detectable siloxanes from tests performed in July and October 2004.

3.4 Baseline Siloxane Treatment Costs

As the RP-1 plant does not currently have siloxane removal equipment, there is no baseline treatment cost. However, the cost of not removing siloxanes is seen in operation and maintenance (O&M) costs on the power generation equipment, including the microturbines and generator engines. During this quarter the gas siloxane concentrations were lower than previous periods and the engines did not need to be taken out of service for maintenance work. However, microturbines are more sensitive to gas quality and the cost of not removing moisture and siloxanes can be seen in the fact that the eight micro-turbines at RP-1 have been out of service. This has resulted in equipment with a capital cost of approximately \$800,000 and an electricity generation capacity of 240 kW not being used.

SECTION 4

Additional Baseline H₂S Data

The baseline data collected for both the biological H₂S removal system and moisture/siloxane removal by Chiller/Sagpack were presented in the Second Quarterly Report. This section presents additional baseline H₂S test data obtained during June through September 2005.

4.1 Digester 4 H₂S Data

The biological H₂S scrubber will be tested on the gas stream from Digester 4. This digester is used to process dairy manure, and ferric chloride is directly added to this digester to control the H₂S in the digester gas produced. Foodwaste addition commenced in April 2005, with occasional addition of salad dressing, lactose and ice cream wastes. The food waste co-digestion with manure is discussed in detail in other reports. Table 4-1 summarizes the monthly average ferric chloride addition to this digester and the average H₂S level in the digester biogas. As shown in this table, ferric chloride added to the manure digester averaged 134 gallons per day (gpd), which is approximately 1,648 lb/d with a 40 percent active solution. H₂S in the biogas ranged from 40.5 to 70.1 ppm with an average of 56.2 ppm. The mass of H₂S produced in the digester gas ranged from 0.40 to 1.03 lb/d with an average of 0.58 lb/d. Assuming the H₂S concentration in the digester gas would be 2,000 ppm without H₂S control, the estimated amount of H₂S removed by ferric injection ranged from 11.9 to 26.7 lb/d, and averaged 18.9 lb/d. Detailed H₂S baseline data is included in Appendix B.

TABLE 4-1
Monthly Average of the Ferric Chloride Addition and H₂S Produced from RP-1 Digester 4

Month	Digester Feed gpd	Feed TS lb/d	Ferric Chloride Added		Gas Produced cfd	H ₂ S Conc. in Dig. 4 Biogas		Theoretical Removal ² lb/d
			gpd	lb/d ¹		ppm	lb/d	
Jan-05	39,330	30,330	108	1,277	113,428	68.1	0.69	20.0
Feb-05	38,970	27,873	124	1,469	101,011	49.7	0.47	17.7
Mar-05	41,638	35,310	101	1,376	106,193	40.5	0.40	18.8
Apr-05	35,054	31,793	99	1,309	110,979	40.5	0.42	19.6
May-05	26,256	36,140	119	1,405	102,097	41.8	0.42	18.0
Jun-05	23,450	30,588	135	1,670	93,223	75.8	0.69	16.2
Jul-05	27,050	36,694	166	2,104	68,461	57.2	0.43	11.9
Aug-05	40,548	58,045	185	2,190	122,806	62.0	0.69	21.6
Sep-05	29,733	37,482	171	2,028	154,207	70.1	1.03	26.7
Average	33,559	36,028	134	1,648	108,045	56.2	0.58	18.9

Note:

¹ Assume ferric chloride is 40 percent active by weight, and the density is 1.4176 g/cm³ (11.8304 lb/gal at 20°C) at 40 percent active.

² Assume H₂S in the biogas would be 2,000 ppm without ferric chloride addition.

Figure 4-1 shows the daily ferric chloride dose expressed as gallons of ferric chloride per gallon of feed to Digester 4 and the biogas H₂S concentration, while Figure 4-2 shows the impact of foodwaste addition on the H₂S production. The data show that addition of foodwaste increased the concentration of H₂S in the digester gas and the dose of ferric chloride also was increased to account for this.

FIGURE 4-1
RP-1 Digester 4 Ferric Dose and Gas H₂S Concentration

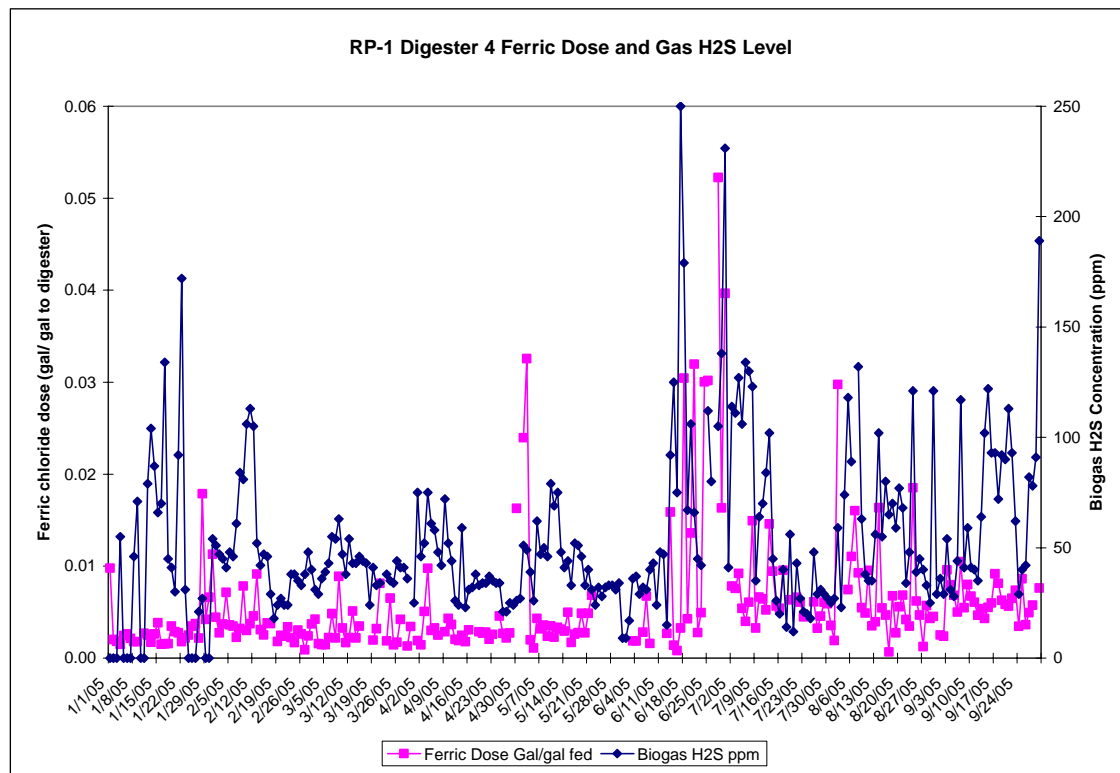
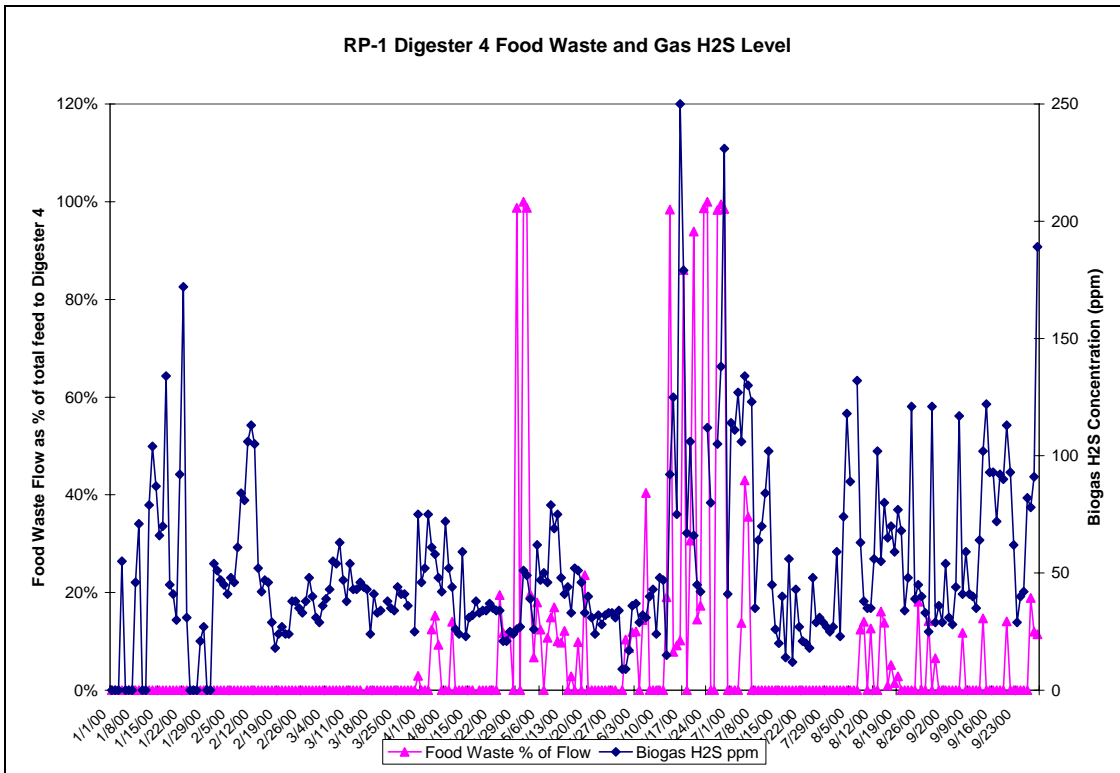


FIGURE 4-2
 RP-1 Digester 4 Food Waste and Gas H₂S Concentration



4.2 Baseline Ferric Chloride Digester 4 Gas Treatment Costs

The cost for purchasing ferric chloride is \$318/ton (includes 7.75 percent tax), according to the plant staff. Using this cost, along with the known amount of ferric chloride added to the digester and other data presented above in Table 4-1, the unit cost of ferric chloride addition is calculated and summarized in Table 4-2. As shown in this table, the average daily cost is \$251, which corresponds to 0.8 cent per gallon of digester feed, 0.7 cent per pound of TS feed, or 0.2 cent per cubic feet biogas generated; and \$14 per pound of H₂S removed from the biogas through addition of ferric chloride, assuming that the H₂S concentration would be 2,000 ppm without treatment.

TABLE 4-2
H₂S Control Costs for Manure Digestion Using Ferric Chloride

Month	Ferric Chloride Cost		Cost per Gallon Digester Feed \$/gal manure	Cost per Unit TS \$/lb TS	Cost per Unit Biogas Produced \$/cf gas	Cost per Unit H ₂ S Removed \$/lb H ₂ S Removed
	\$/month	\$/day				
Jan-05	6,258	202	0.005	0.007	0.002	10
Feb-05	6,499	232	0.006	0.008	0.002	13
Mar-05	5,874	189	0.005	0.005	0.002	10
Apr-05	5,565	186	0.005	0.006	0.002	9
May-05	6,884	222	0.008	0.006	0.002	12
Jun-05	7,560	252	0.011	0.008	0.003	16
Jul-05	9,597	310	0.011	0.008	0.005	26
Aug-05	10,730	346	0.009	0.006	0.003	16
Sep-05	9,614	320	0.011	0.009	0.002	12
Average	\$7,620	\$251	\$0.008	\$0.007	\$0.002	\$14

Next Steps—Gas Cleaning Pilot Test Phase

During the next phase of the test, the gas cleaning pilot phase, the installed gas drying, biological H₂S removal, and packaged siloxane removal systems will be tested. The chiller and Sagpack™ equipment will be re-started once the treated gas line is re-routed to the generator engines.

5.1 Equipment and Instrumentation to Conduct Test

The equipment required to collect a biogas sample is a Tedlar bag or a field test kit, such as a canister or impinger, supplied by the laboratory where the testing will be conducted. Each of the sample ports will require a manual valve to fill the biogas container for the sample. Quotes will be obtained from laboratories with the capability to test for siloxanes, moisture, H₂S, and other common biogas constituents. A power meter with totalizer will be used to measure the electricity consumed by each of the systems. The reading and recording of this parameter will be done manually. Pressure gauges and thermowells will be used to measure the pressure and temperature of the biogas. The reading and recording of these parameters will be done manually. A thermal mass flowmeter with totalizer will be used to measure the flow of biogas. The reading and recording of this parameter will be done manually.

5.2 Test Procedures

Biogas testing needs to be performed once a week, or bi-weekly as required for each of the pilot testing equipment. The procedure will be as follows: a biogas sample will be collected at each of the sample ports and sent to the laboratory for testing. The laboratory will perform three primary tests – EPA TO-15 to test for volatile organics and siloxanes, EPA/CARB 16 for sulfur species, and moisture. The test results will be recorded. It is expected that the biogas samples will be collected by personnel from the RP-1.

For all pilot systems, the testing program also will include daily monitoring and recording of biogas flow, temperature and pressure, and equipment power consumption.

The testing program for the gas drying and the H₂S scrubber will require weekly collection and analysis of the condensate as well as recording of these results. The condensate flow from the drying equipment will be monitored and recorded daily from a flowmeter installed on the condensate line. The temperature of the condensate will be measured and recorded on a regular basis.

Table 5-1 contains a summary of the recommended sample collection, tests, data monitoring, and data recording to be performed, and a frequency schedule for each.

TABLE 5-1
Sampling Plan

Sample/Parameter Monitored	Test	Frequency
Gas Drying System		
Biogas upstream of equipment	Moisture/siloxane/VOCs	Biweekly for months 1 and 2, weekly for month 3
Biogas downstream of equipment	Moisture/siloxane/VOCs	Biweekly for months 1 and 2, weekly for month 3
Temperature	--	Once a day
Pressure	--	Once a day
Flow	--	Once a day
System power consumption	--	Once a day
Condensate	Volume and temperature	Once a day
Condensate	Composition (VOC, NH ₃ -N, total sulfur, TS, TDS, and pH)	Once a month
Siloxane Saggpack System		
Biogas downstream of HOX unit	Moisture/siloxane/VOCs	Biweekly for months 1 and 2, weekly for month 3
Biogas downstream of Graphite unit	Moisture/siloxane/VOCs	Biweekly for months 1 and 2, weekly for month 3
Temperature	--	Once a day
Pressure	--	Once a day
Flow	--	Once a day
Biological H₂S Removal System		
Biogas upstream of equipment	Reduced sulfur compound	Once a week
Biogas downstream of equipment	Reduced sulfur compound	Once a week
Temperature	--	Once a day
Pressure	--	Once a day
Flow	--	Once a day
System power consumption	--	Once a day
Scrubber overflow	Volume and temperature	Once a day
	Composition (VOC, NH ₃ -N, total sulfur, TS, TDS and pH)	Once a week

Each equipment system used in this pilot testing program will be provided with the necessary controls and monitoring instrumentation for proper and safe operation. The refrigerated dryer system contains the controls for the refrigeration cycle. The Saggpack siloxane removal system uses media and does not require control.

The biological H₂S removal system is provided with equipment, instrumentation, and appurtenances to control the process. The air compressor supplying the necessary air to the process is controlled by an oxygen sensor. If the oxygen sensor reaches the high level setpoint, the air compressor stops operation. The pH of the fluid is also measured and makeup water will be added to the tank if the pH sensor reaches the low setpoint. The overflow

system will allow excess condensate to drain. Dräger-tubes will be used for local measurement of H₂S.

The laboratory must follow the equipment calibration recommendations to meet the ASTM and EPA requirements for the tests that are required above. Pressure gauges, thermowells and power meters will require calibration every year. Thermal mass flowmeters will require calibration every 3 months.

Other data that will be collected are the cost of supplies for each system and the labor hours dedicated to the operation and maintenance of each system. The time to inspect instruments and collect and record parameters will be documented separately for each system.

5.3 Data Analysis Procedures

The data will be input into an Excel spreadsheet containing the necessary calculations for the required analysis. The expected calculations are removal percentages for H₂S, moisture, and siloxanes. Another Excel spreadsheet will be created with the raw data and the calculations to determine the operational cost for each pilot system.

SECTION 6

Quality Assurance and Data Analysis Procedures

This section describes quality assurance (QA) and data analysis procedures followed for the data collected through the fifth quarter.

6.1 Quality Assurance Procedures

Two important steps in practicing quality assurance and control (QA/QC) were performed. The first step ensured good sample collection and shipping methods. Prior to data collection, the appropriate sample collection points and collection times were demonstrated to the plant operators. The samples were collected by the operators in appropriate bottles provided by the laboratory that was conducting the tests. For samples sent to an external laboratory, the sample bottles were stored on ice and shipped in coolers on the same day if possible, or before 10:00 a.m. the next morning for samples taken later in the day. When same day shipping is not possible (usually weekends and evenings), the sample bottles were preserved, stored (usually at 4°C), and shipped the next morning to ensure that the appropriate procedures and holding times are met, as specified by the analytical laboratory.

The second step relates to laboratory procedures and methods. During laboratory analyses conducted by the plant's certified laboratory or by an external certified laboratory, laboratory staff demonstrated familiarity with standard sample storage, analysis, and QA/QC procedures. When immediate analysis was not possible (usually weekends and nights), the sample bottles were preserved, stored (usually at 4°C), and delivered to and analyzed in accordance with appropriate procedures and holding times, as specified by the analytical laboratory. Replicate samples and split sample analysis were conducted occasionally to verify reliability of results.

At a minimum, the laboratories' QA programs are required to meet EPA and ASTM standard. In the field, the collection of the condensate samples was done using sterilized containers. Immediately after collecting the sample, the container was labeled with the sample number, source, date, and time.

6.2 Data QA/QC and Analysis Procedures

In addition to the QA/QC of the sample preparation and laboratory analysis procedures, QA/QC and data analysis of the collected data were critical to an accurate representation of gas cleaning system performance. This included development of a daily log sheet for recording operational parameters of the chiller, SagPack and biological scrubber systems. Information from the log sheets is in a format that can be easily input into an Excel spreadsheet. On-line data collected by the plant, such as flow rates, and laboratory data were input into Excel spreadsheets.

For the gas cleaning baseline data, an Excel template was developed to host and analyze the historical monthly operational and performance data obtained from the plant for both the sludge and manure digestion systems. The summary of the monthly data from these files will be input into an Excel spreadsheet to do the calculations, such as H₂S removal percentages by ferric chloride addition and iron sponge system, and operational costs. Excel spreadsheets were also created to host the two rounds of laboratory test results.

APPENDIX A

Siloxane Sampling Protocol

APPENDIX B
Baseline Gas Cleaning Data
