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Impact and Abatement of Siloxanes in the Bucklin Point WWTF Anaerobic Digestion Process

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**IMPACT AND ABATEMENT OF SILOXANES IN
THE BUCKLIN POINT WWTF ANAEROBIC
DIGESTION PROCESS**

**BY
BARRY WENSKOWICZ**

**A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF
MASTER OF SCIENCE
IN
CHEMICAL ENGINEERING**

UNIVERSITY OF RHODE ISLAND

2011

MASTER OF CHEMICAL ENGINEERING THESIS
OF
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ABSTRACT

The biogas produced at the Bucklin Point Wastewater Treatment Facility contained a high concentration of siloxane compounds. A manufacturer of personal care products was found to be the major source of the siloxanes. Much of the siloxane that the manufacturer discharged into the sewage collection system entered the anaerobic digestion system, where a portion was released into the biogas depending on the siloxane concentration and the amount of volatile solids that were digested. All of the siloxanes in the biogas likely oxidized to silica when the biogas was combusted, and a portion of the silica deposited in the boiler and impeded heat transfer efficiency by impacting the overall heat transfer coefficient. Siloxanes were condensed from the biogas at 34°F and the resulting concentrations were comparable to the concentrations predicted by theory.

ACKNOWLEDGMENTS

I thankfully acknowledge my employer the Narragansett Bay Commission, my Supervisor Jim McCaughey, my Academic Advisor Dr. Stanley Barnett and my wife Diane Wenskowicz for their support.

PREFACE

This work is the result of research on the high siloxane loadings at the Bucklin Point Wastewater Treatment Facility (WWTF) that is located in East Providence, Rhode Island and is owned by the Narragansett Bay Commission (NBC). Most of the information was collected from 2005 through 2008 and is presented here in manuscript format. The research was supported by the NBC because the resulting information is potentially useful for assessing methods to regulated siloxane discharges and to assess methods to fully utilize the biogas. Specifically, the amount of siloxane in wastewater would need to be known in order to possibly apply a mass-based surcharge fee or to possibly enforce local discharge limit for siloxanes. In addition, siloxane's impact on the WWTF operations would need to be known to determine a monetary penalty and to determine if it is economic to clean the biogas.

Much of the information in this study was presented at the 2010 Annual Conference of the New England Wastewater Association in Boston, Massachusetts on January 28, 2010. An article comprised of the manuscript, but not including Chapter - 2 Review of Literature (pages 5 and 6), was published in the spring 2010 Edition of the NEWEA Journal (volume 44, No. 1, page 24 to 34).

The study is unique because a relatively large amount of siloxane was discharged into the NBC sewage collection system compared to the amount normally discharged by residential sources. A major manufacturer was found to be responsible for the large amount. This study proposes the hypothesis that absorbed siloxanes are released into biogas as a result of the anaerobic digestion of siloxane-bearing bio-solids. The

hypothesis was tested by determining the amount of siloxane entering and leaving the digester at high and low siloxane loading when the volatile solids reduction was approximately 50%.

This study proposes the hypothesis that utilizing siloxane-bearing biogas causes a silica deposit to form in the boiler that impedes heat transfer and reduces the recovery of thermal energy. The basis for this hypothesis is that common soot is known to impeded heat transfer in boilers and silica is 10 times more resistant to heat transfer than carbon soot. The hypothesis was tested by determining boiler efficiency and the heat transfer rate at high and low siloxane loading.

The most widely used method to remove siloxanes from biogas is to dry it and then pass it through an adsorptive bed of disposable granular activated carbon (GAC) media. During drying, a biogas chiller may be used to remove the water vapor. Some siloxane may condense with the water depending on the type of siloxane, its initial concentration and the final biogas temperature. This study proposes the hypothesis that the amount of siloxane that condenses can be predicted using thermodynamic data and theory. The hypothesis was tested by operating a bench-scale condenser at 33°F and comparing the results to theory.

TABLE OF CONTENTS

ABSTRACT.....	ii
ACKNOWLEDGMENTS.....	iii
PREFACE.....	iv
TABLE OF CONTENTS	vi
LIST FIGURES.....	viii
CHAPTER 1	1
INTRODUCTION.....	1
CHAPTER 2	5
REVIEW OF LITERATURE.....	5
CHAPTER 3	7
AMOUNT, IMPACT AND CONDENSATION OF SILOXANE	7
CHAPTER 4.....	50
CONCLUSIONS	50
APPENDIX 1 - PARTITION COEFFICIENTS.....	51
APPENDIX 2 - SILICA REMOVED FROM BOILERS.....	53
APPENDIX 3 - SILICA DEPOSIT DENSITY	58
APPENDIX 4 - BOILER HEAT TRANSFER RATE.....	60
APPENDIX 5 - EQUIPMENT SPECIFICATIONS.....	62
APPENDIX 6 - BIOGAS SAMPLE VOLME DETERMINATIONS.....	70
APPENDIX 7 - TS AND VS ANALYTICAL PROCEDURE.....	82
APPENDIX 8 - TSS AND CVSS ANALYTICAL PROCEDURE	86

APPENDIX 9 - SILOXANE ANALYTICAL PROCEDURE	89
APPENDIX 10 - COMMERCIAL LAB COA BY DATE.....	99
BIBLIOGRAPHY	229

LIST OF FIGURES

Figure	Caption	Page
Figure 1	Biogas, Boiler and Digester Heat	3
Figure 2	Physical Properties of Siloxane Compounds	7
Figure 3	Bucklin Point Silicon Balance	8
Figure 4	Digester Influent D5 Partition Coefficient	12
Figure 5	D5 Siloxane in Digester Sludge	13
Figure 6	Potential Siloxane in Digester Influent	15
Figure 7	Volatization in the Collection System	17
Figure 8	Siloxane from Manufacturer during Slug Loading	19
Figure 9	Maximum Values during Slug Loading	20
Figure 10	Average Siloxane from Manufacturer during 3 Year Timer Period	22
Figure 11	Siloxane in Biogas during 3 Year Time Period	23
Figure 12	Siloxane Removed from Boilers during 3 Year Time Period	24
Figure 13	Historic Average Values	25
Figure 14	Current Normal Values	27
Figure 15	Siloxane Volatization	27
Figure 16	Silica Deposition	29
Figure 17	Bucklin Point Digester Heat Balance	31
Figure 18	Deposit Impact on Stack Gas Temperature Measurement	32
Figure 19	Corrected and Uncorrected 2009 Stack Gas Temperature Measurement	33
Figure 20	Corrected and Uncorrected 2008 Stack Gas Temperature Measurement	34
Figure 21	Boiler Efficiency during 2008 and 2009	37
Figure 22	Values used to Determine Boiler Heat Transfer during June 2008	39
Figure 23	Boiler Heat Transfer as a Function of Deposition	40
Figure 24	Contributions to Boiler Overall Heat Transfer Resistance	41
Figure 25	Biogas Content and Characteristics	42
Figure 26	Pilot Chiller	44
Figure 27	Pilot Chiller and Condensate Trap	46
Figure 28	Pilot Chiller Results	46
Figure 29	Comparison of Actual and Theoretical Siloxane Partial Pressure	47

CHAPTER 1

INTRODUCTION

The anaerobic digestion operation at the Narragansett Bay Commission's Bucklin Point Wastewater Treatment Facility (WWTF) was seriously affected by siloxanes that originated primarily from a manufacturer of personal care products that was in operation until October of 2008.

The concentration of siloxane measured in the biogas produced at the Bucklin Point WWTF reached a concentration of 62 parts per million by volume (ppmv). This is 30 times more than the amount found at most WWTFs where the concentrations average approximately 2 ppmv (Wheless, 2002). The excessive amount of siloxanes hindered efforts to use more of the biogas in a proposed combined heat and power (CHP) system by sharply increasing potential operating and maintenance costs.

As the facility dealt with these issues over several years, it addressed many important questions, including how much siloxane was in the wastewater and biogas, the impact of the resulting silica on the boilers, and whether it was necessary to clean up the biogas using a condenser followed by a carbon adsorption system to fully use the biogas in a low-maintenance CHP system.

OPERATIONS

The Bucklin Point WWTF is in East Providence, R.I., and treats an average of 24 million gallons per day (MGD) of industrial and residential wastewater as well as a small amount of storm water. It is an advanced and efficient treatment facility.

The solids collected during the treatment process include grit, hauled septage, primary sludge, waste activated sludge and floating scum removed in the primary clarifiers. All solids except grit are processed in three anaerobic digesters where they are mixed, heated and allowed to decompose. The mesophilic anaerobic digestion process converts over half the volatile solids into a valuable and renewable biogas fuel, containing over 60 percent methane and having a heat value of 600 Btu per standard cubic foot (SCF) of volume. This natural biological process cuts in half the volume of solids that need to be shipped offsite where they are land-applied or incinerated.

Much of the biogas produced is burned in boilers to heat the digesters. Any excess biogas that cannot be used is burned in a flare to convert the methane (a strong greenhouse gas [GHG]) into carbon dioxide (a weaker GHG). Properly managing sludge biosolids causes GHG emissions that are generally considered biogenic. Using energy recovered from the renewable biogas offsets GHG emissions that otherwise typically would be produced by burning a non-renewable fossil fuel.

For at least 10 years, WWTF personnel have been aware that a white residue forms whenever the biogas is burned in a boiler or in a flare, and have regularly removed it from the boilers. The facility also uses natural gas to help heat the digesters when needed. Operating personnel continually strive to maximize biogas use and minimize natural gas use.

Biogas or natural gas is burned in one or two of the three relatively new boilers to produce hot water used to heat the digester's biosolids. Each boiler is rated for 3.11 million (MM) Btu/hr, which is the approximate maximum wintertime heat demand for the entire digester system.

The new boilers were designed and installed in 2004 with a fouling allowance so they could burn siloxane-contaminated biogas while still delivering enough heat to meet the demand of the digesters. Additionally, their heat transfer tubes were oversized to a 3-inch diameter to make it easier to remove the accumulated silica.

Figure 1. Biogas, boiler and digester heat

The digesters produce an average of over 10,000 SCFH or 6 MMBtu/hr of biogas as shown in Figure 1, which is typical based on the population served. The biogas production rate varies seasonally. It dips in the summer and peaks in the late winter even though the temperature in the digester always remains at 95°F. The average measured

biogas production rate is within 6 percent of the rate predicted by a mass balance of volatile solids entering and leaving the digester. There were no measurements of biogas production while the biogas pipelines were cleaned and new meters installed between the summers of 2006 and 2007.

The amount of heat transferred into the digesters is shown by the lower dotted line in Figure 1, where the high winter demand peaks are easily discerned. More than enough biogas is usually produced (shown by the upper solid line) to provide the input needed by the boiler (shown by the middle dashed line) to heat the biosolids and maintain the temperature. A large difference in heat value between the boiler feed and the heat transferred indicates heat is being wasted due to inefficient heat transfer. A small difference such as during the spring of 2009 indicates the heating system is relatively efficient. Biogas that cannot be used is flared. On Figure 1, the flared amount is the difference between the upper solid line and the middle dashed line, adjusted for natural gas use.

CHAPTER 2

Review of Literature

During the past 10 years there has been a large amount of research published on siloxanes in biogas from wastewater treatment facilities with a focus adsorptive methods to remove siloxanes from biogas and on the analytical techniques to identify the siloxanes. Interest in the field has been prompted by an increased desire to use renewable fuels efficiently in combined heat and power systems because clean renewable fuel can be used to economically produce useful heat and power that offsets fossil fuel use. The relatively recent introduction of micro-turbines to the market, the availability of renewable energy credits and increasing natural gas prices have helped spur interest in this area.

A small amount of research has been published related to the behavior of siloxanes at wastewater treatment facilities that use anaerobic digestion systems. Kaj (et. al. 2004 and 2005) determined the siloxane concentration in many environmental matrices including WWTF influent and digested sludge. Parker (1999) studied the fate of D4 and D5 siloxanes in a pilot-scale WWTF without anaerobic digestion and did not determine the amount of siloxane in scum. Oshita (et.al. 2007) found that 20% to 50% of the D5 that entered an anaerobic digester moved into the biogas that was formed. Siloxane is in a class of compounds that are considered emerging micro-pollutants that are receiving increased attention recently.

No published research is available on the impact of silica on boiler heat transfer even though this type of impact may apply to all systems that recover thermal energy from biogas. One reason for this may be that renewable energy credits are not typically available for generating renewable heat energy. New interest in carbon foot-printing may

spur more interest in this area. Lin (2008) has determined thermophoretic particle deposition efficiencies that may apply to silica. Cruz (2009) has determined the effect of an applied voltage on the deposition rate of soot. Lee (et. al. 2001) reported that silica deposited inside the boiler. Much has been published regarding silica aerogels but these compounds have a much lower density, conductivity and melting point than the silica in this study.

Some research has been published on the removal of siloxanes by condensation or freezing and a large amount has been published on their removal by other methods such as adsorption and absorption. Condensation is less effective at the low siloxane concentrations and few processes are fully developed commercially. Oshita (et. al. 2010) studied the removal of siloxane from biogas using activated carbon and found that the siloxane molecules are generally larger than micropores and absorbents with pores of relatively large diameters are desirable. Wheless and Jeffery (2004) found that the Selexol™ process removed 99% of the siloxane. Rossol (et. al. 2003) found that deep chilling is economical only at elevated siloxane load. McCarthy (et. al. 2007) also reported on the cryogenic siloxane removal process. Ajhar (et. al. 2010) reported theoretically attainable siloxane concentrations and removal efficiencies by condensation using Aspen™ software. A patent held by Olsen (2008) involves expanding the biogas to cool it then removing the siloxanes and ice using a cyclonic separator. A 2-step Exilox process involves cooling the biogas (Muche 2000) to 5°C to remove most of the water vapor and deep-chilling it to -30°C to remove residual condensable contaminants. Palczewska (et. al. 2005) and Colona (et. al. 2006) experimentally determined saturation vapor pressures for pure D4 and D5 siloxanes.

CHAPTER 3

Amount, Impact and Condensation of Siloxanes

Siloxane & Silica Flows – Mass Balance

Waste streams throughout Narragansett Bay Commission's Bucklin Point (BP) sewage collection and treatment system were analyzed for siloxanes after silica was found to interfere with sludge boilers at the facility. A summary of the analyses by commercial laboratories can be found in Appendix 1. Determining the amount of siloxane that caused the interference would allow the wastewater treatment facility (WWTF) to anticipate impacts based on the weight of siloxane entering the facility and plan accordingly. Once the cost of the impact is known then a penalty against responsible parties could be determined based on the weight discharged.

The following volatile siloxanes were analyzed at various times and at several locations in the sewage collection and treatment system:

Physical Properties of Siloxane Compounds											
Siloxane Compound	Synonyms		MW	MP (F)	BP (F)	SpGr	LogK _{ow}	Vapor Pressure (mmHg)	Solubility (mg/l)	Henry's Constant (-)	CAS
Hexamethyldisiloxane	MM	L2	162				4.20	42	0.930	397	
octamethyltrisiloxane	MDM	L3	237					3.9	0.034	1465	
decamethyltetrasiloxane	MD2M	L4	311					0.4	0.007	943	
dodecamethylpentasiloxane	MD3M	L5	385								
tertadecamethylhexasiloxane		L6	484								
Hexamethylcyclotridisiloxane		D3	222	148	275	1.02	3.85	8.6	1.560	-	
Octamethylcyclotetrasiloxane	OMCTS	D4	297	64	348	0.96	4.45	1.0	0.056	259	556-67-2
Decamethylcyclopentasiloxane	Cyclomethicone	D5	371	-36	412	0.96	5.20	0.2	0.017	185	541-02-6
Dodecamethylcyclohexasiloxane		D6	444			0.97	5.86	0.0	0.005	104	

Figure 2. Physical Properties of Siloxane Compounds

Only D4 and D5 siloxanes were ever found in significant amounts. The term “siloxanes” is used in this study to refer to D4 and D5. Normal background levels of these compounds have been studied at other wastewater treatment facilities and their fate within a pilot-scale wastewater treatment system without anaerobic digestion has been studied (Parker, 1999). However, no study has been published on the impact of elevated siloxane concentrations on sludge boilers at a WWTF.

The flow of cyclic semi-volatile D4 and D5 siloxane compounds through the collection and treatment system is shown in the following diagram:

Figure 3. Bucklin Point Silicon Balance

When siloxane first enters the sewage collection system it is known to strongly adsorb onto the organic solids. Incoming wastewaters from the Blackstone Valley Interceptor

(BVI) and the East Providence Interceptor (EPI) combine at the head works of the BP WWTF and then pass through a vortex separator where heavy grit is removed. The wastewater then passes through primary clarifiers where 54% to 73% of the total suspended solids are removed. It is estimated, based on the work of Parker, that 94% of the siloxane from the primary settled solids is transferred into the anaerobic digester (the balance evaporates). Any floating organic scum that may be present in the primary clarifier is automatically removed by skimming and is transferred into the digester separately.

Wastewater containing unsettled solids leaves the primary clarifiers and enters the secondary aeration basins where solids and activated sludge are aerated and much of the siloxane becomes stripped into the atmosphere. Waste activated sludge (WAS) from the secondary aeration basin is thickened in a dissolved air flotation (DAF) system where siloxane also becomes stripped into the atmosphere. It is estimated that less than 10% of the siloxanes initially adsorbed on secondary solids is transferred into the anaerobic digester based on Parker's pilot study.

The WAS and primary sludge are mixed and heated in the digester. Approximately half the volatile solids in the sludge are biologically degraded into methane and carbon dioxide (biogas) over the course of an 18 to 26 day detention time. During this time siloxanes are released from the digesting solids and enter the biogas. This study

proposes that the reduction of the volatile solid substrate in the digester is the major driver that affects the release of siloxane into biogas.

Methodology

Samples of the wastewater, sludge, biogas and boiler silica were collected at different times over a 3 year period from select locations shown in the mass balance diagram.

The locations were chosen included a major manufacturer, WWTF head works, anaerobic digester influent, biogas delivery system and boiler. The samples were analyzed using various techniques described in the appendix. All siloxane samples were collected with zero headspace. Samples that were to have the aqueous phase analyzed were not refrigerated and were centrifuged immediately prior to analysis to remove suspended solids from the liquid phase.

The wastewater samples that were collected from the manufacturer, head works and digester influent during a one week period in 2007 were analyzed by a commercial laboratory to determine the siloxane concentrations in the aqueous and sludge phases. The lab used an extractive analytical technique (Dewil 2007) that was also used in the pilot study by Parker.

The digester influent was sampled to determine the sorption characteristics of sludge. The manufacturer and head works were sampled to determine if the manufacturer was a significant source of siloxanes that entered the biogas.

Digester Sludge Sorption

The sorption coefficient is a measure of the extent that siloxane adsorbs onto the volatile organic solids (VSS) in the sludge. The coefficient is a characteristic of the type of sludge and total concentration of volatile solids in the wastewater. It is defined in part by the concentration of siloxane in the liquid phase and the total concentration which includes siloxane adsorbed onto sludge solids.

These concentrations were determined to establish the sorption coefficient for D5 siloxane in the digester sludge at the BP WWTF. D5 was the predominant siloxane found in the sludge. NBC wanted to know this value so that the amount of siloxane entering the digester could possibly be predicted based on liquid phase concentration.

Methodology

The total concentration of siloxane in a sample of digester sludge was determined by analyzing a raw sample of sludge collected on May 29, 2007. The sample was comprised of equal volumes of thickened waste activated sludge and primary sludge. A portion of the sample at room temperature was centrifuged at 5,000 rpm for 5 minutes and the solids-free supernatant was analyzed to determine the concentration of siloxane in the liquid phase. The maximum water solubility of D5 siloxane is 0.017 mg/l. Visual observation of the decant showed it was turbid which indicated a portion of the siloxane may be emulsified as a fine colloid.

Findings

The following total and aqueous phase D5 concentrations were determined and used to calculate the sorption coefficient:

Digester Influent D5 Partition Coefficient		
Sample Matrix	Concentration (mg/l)	K _P (L/g)
Aqueous	0.079	29
Total	75	

Figure 4. Digester Influent D5 Partition Coefficient

A total of 46.5 lb/day of siloxane entered the digester on 5/29/07 based on the 75 mg/l concentration and the measured sludge flow rate of 74,264 gpd. The certificate of analysis for this result from Atlantic Analytical Laboratory can be found in the Appendix. The mass flow rate of siloxane in the waste streams was calculated from the following equation:

$$M_{\text{siloxane}} = F1 \times C \times Q_{\text{wastestream}}$$

Equation 1

where M_{siloxane} is the mass of siloxane in units of pounds per day, C is the concentration of siloxane in the waste steam in milligrams per liter, Q is the waste stream flow rate in units of gallons per day and F1 is a conversion factor in units of (lb x liter) / (mg x gal).

The D5 sorption coefficient of 29 was calculated based on the following equation:

$$C_t = C_s + K_p (X) (C_s)$$

Equation 2

where K_p is the adsorption coefficient in units of L/g, and C_t , C_s and X are the total concentration, dissolved concentration and the total volatile suspended solids (VSS) concentration, respectively in g/L. The concentration of volatile solids in the sludge was 33,125 mg/l. The coefficient can be calculated directly by rearranging the above equation as follows:

$$(C_t/C_s - 1) = K_p (X)$$

Equation 3

The concentration in the sample (square data point) is plotted with the concentrations based on the work of Parker for each individual sludge type:

Figure 5. D5 Siloxane in Digester Sludge

The sorption coefficients are proportional to the slope of the lines plotted in the diagram. The results indicate that the 29 L/g sorption coefficient for D5 in the BP digester sludge (square data point) is lower than the average coefficient of 53 L/g determined in the pilot study (solid black line).

At a sorption coefficient of 29 L/g and a volatile solids concentration of 33.1 g/l, there is 949 units of mass absorbed on the sludge for every unit of mass in the liquid phase. It is important to note that the C_s in BP sludge included dissolved as well as emulsified D5 and the mechanism of its adsorption on primary solids is not well understood.

It is possible that the sorption coefficient was lower than expected because the sludge may have been near saturation where an increase in C_s may no longer cause more adsorption but may result in emulsification of D5 in the liquid phase. Such behavior could lead asymptotically to a horizontal line in the plotted curve (not shown). It is also possible that near saturation the D5 is loosely held in layers around the sludge.

Digester Scum

The 46.5 lb/day of siloxane found in the sludge entering the digester on 5/29/07 can be used to back-calculate the amount of siloxane entering the primary clarifier. It should be noted that scum enters the digester separately but was not analyzed.

Potential Siloxane in Digester Influent					
Waste Stream	Total Concentration (ug/l)	Aqueous Concentration (ug/l)	Kp (l/g) ¹	Flow (gpd)	Loading (lb/day)
Sludge _{actual}	75,000	79	24.2	74,264	46.5
Sludge _{theoretical}	137,465	79	52.5	74,264	85.2
Scum _{theoretical}	12,520,656	79	5.20	923	96.4

Note¹: D5 Log Octanol/Water coefficient is listed for scum

Figure 6. Potential Siloxane in Digester Influent

The total concentration of siloxanes in the sludge was 75 mg/l. At a sludge flow, rate of 74,264 gpd this equates to a 46.5 lb/day of siloxanes that entered the digester. Primary clarifier TSS removal was 54% on 5/29/07 and an estimated 94% of the siloxane initially in primary sludge was transferred into the digester. The secondary clarifier TSS removal was 46% and an estimated 5% of the siloxane initially in the secondary sludge was transferred into the digester. Based on this information, the total amount of siloxane entering the digester can be expressed in terms of the amount of siloxane entering the primary clarifier (PC_{in}):

$$(0.94*0.54 + 0.05*0.46)*PC_{in} = 46.5 \text{ lb/day}$$

Equation 4

The equation can be solved for PC_{in}, yielding 87.6 lb/day of siloxane entering the primary clarifier and WWTF on 5/29/07.

It is possible that the scum could have carried additional siloxane into the digester that is not included in the 87.6 lb/day. The scum could have contained up to 12,521 mg/l if it

was in equilibrium with the sampled sludge based on the D5 siloxane octanol-water partition coefficient of 5.2. At a scum flow rate of 923 gpd, this amounts to an extra 96.4 lb/day that brings the total maximum potential to 184 lb/day.

Although this is possible, it is more likely that the scum carried less than the equilibrium value. For example, if it carried only 38.7 lb/day ($85.2 - 46.5 = 38.7$) of siloxane that may have separated from the sludge in the vortex separator this would account for the lower than expected C_t found in the digester sludge on 5/29/07.

Maximum Siloxane Flow

Methodology

The siloxane loading was also unexpectedly high when the wastewater samples were collected from the manufacturer and head works on June 7, 2007. The results from Atlantic Analytical Laboratory are summarized in Appendix 2. The concentrations that were measured during this time period were the maximum that have been observed at the facility and are considered slug loads that are intermittently much higher than any normal variation in the concentration.

Findings

Using the previously defined equation for mass flow, the results from 6/7/07 indicate that the manufacturer discharged 329 lb/day of siloxane and 446 lb/day of siloxane entered the WWTF through the Blackstone Valley Interceptor. Values used to calculate the mass flow are listed in Figure 9.

If residences discharged an estimated 2.96 lb/day of siloxane, based on a normal influent concentration of 15 ug/l (see Figure 14) and little or no siloxane evaporates in the collection system, then the WWTF influent would be expected to contain 331 lb/day assuming there is no accumulation within the collection system. The influent value is adjusted to account for the small amount of siloxane lost to volatilization and the added amount from residential sources. However, the WWTF influent contained 446 lb/day which is 34% higher than the amount estimated based on discharges from residences and the manufacturer.

The following figure shows that it is valid to assume that amount of siloxane that volatilized in the collection system is small based on the Henry's coefficient (Gary, 2002 and Kochetkov, 1999) and an estimated gas flow rate:

Volatization in the Collection System							
Waste Stream	Siloxane Concentration in Wastewater (ug/l)	WW Temp (F)	Henry's Constant (atm*m3/mol)	Equilibrium Gas Phase (ppmv)	Gas/Liquid Ratio	Estimated Gas Phase Concentration (ppmv)	Amount Volatized (lb/day)
WWTF Influent	15	55	0.127	5.14	1.0	0.056	0.18
Digester Influent	79	95	0.833	178	18.0	35	8.3
WWTF Influent	97	55	0.127	33.3	1.0	0.37	1.2

Henry's Constant = 0.410 atm*m3/mol at 25 C (Gary 2002)
Gas Flow (gpd) = 23,640,004 GPD
MW D5 Siloxane = 370.8 lb/lbmol

Figure 7. Volatization in the Collection System

These estimated values indicate an average of 0.18 lb/day, and a maximum of 1.2 lb/day volatilized from the collection system during the 3 year period when the manufacturer

was open. This is a small amount compared to the amount present in the wastewater. These estimates are based on the amount that is known to volatilize in the digester where an average of 35 ppmv was measured.

Only a low amount of D5 transfers out of the wastewater at 55F due to the relatively low volatility, high sorption coefficient and a potentially low diffusivity in water. This could be confirmed using a computer model. However, for the purposes of this study the amount volatilized in the collection system at 55 F will be assumed to be small compared to the amount present in the wastewater.

Based on these results, it is most likely that the manufacturer was the sole source of essentially all of the 450 lb/day of siloxane found at the head works. This is based on the fact that no other significant source of siloxane discharge has ever been identified in the district that would account for a significant amount discharged despite an extensive search by the NBC's regulatory Pretreatment section.

The discrepancy in the amounts found at the manufacturer and at the head works may be due to a floating phase that possibly escaped collection at the manufacturer or due to an inaccurate 24-hour composite sample lag time of 3.5 hours that was used to allow the manufacturer's wastewater to reach the WWTF.

During the approximate 2 month time period when slug loading was known to occur, the average amount of siloxane discharged by the manufacturer was 30.7 lb/day based on the following wastewater screening results conducted by NBC:

Siloxane from Manufacturer (Slug Load Months)			
Sample Date	Manufacturer Flow (GPD)	Siloxane Concentration (ug/l)	Siloxane Flow (lb/day)
03/19/07	70,190	52,054	30.5
03/20/07	90,560	42,764	32.3
03/21/07	91,370	24,041	18.330
03/22/07	112,960	37,084	35.0
03/23/07	47,760	49,841	19.9
06/04/07	72,900	40,712	24.8
06/05/07	87,480	44,461	32.5
06/06/07	113,410	46,611	44.1
06/07/07	118,730	39,221	38.9
Average	89,484	41,865	30.7

Figure 8. Siloxane from Manufacturer during Slug Loading

The screening analysis of the wastewater samples is a simplified analytical procedure developed by the NBC laboratory and normalized to certified analyses by Atlantic Analytical Laboratories (the analytical screening procedure, normalization curve and instrument specifications can be found in the Appendix). The results produced are only qualitative due to the difficulty of obtaining repeatable recovery of the D4 and D5 compounds that are essentially water insoluble. Screening results were used when no other analytical results were available. The screening results were used extensively to survey key manholes during the search for other sources of siloxane discharged into the collection system.

The amount of deposit that was removed from the boilers was weighed and recorded each time they were cleaned. During this time period, an average of 3.1 lb/day of predominantly silica-based material was removed from the boilers.

The following figure summarizes the all of concentrations, flow rates and loadings that were determined during the 2 month time period when slug loading was known to occur:

Maximum Slug Values								
Waste Stream Location	Time Period		Concentration		Flow Rate		lb/day	WWTF _{inf} as Silicon
Manufacturer (2 month average siloxane) ¹	3/19/07	6/7/07	155	mg/l	89,484	GPD	30.7	12.29
Manufacturer (siloxane) ²	6/6/07 11:00	6/7/07 11:00	340,000	ug/l	116,070	GPD	329	125
BVI Influent (siloxane) ²	6/6/07 14:30	6/7/07 14:30	2,770	ug/l	19,303,823	MGD	446	169
Boiler (deposit recovered) ³	04/06/07	06/25/07	75.5	%	3,898	SCFH	3.11	1.10

Note¹: Concentration is from screening of manufacturer's discharge by NBC for D4 and D5

Note²: Concentration is from analysis of wastewater by Atlantic Analytical Laboratory for D3, D4, D5 and D6

Note³: Deposit weight measured by NBC

Figure 9. Maximum Values during Slug Loading

The results indicate that of the 12.29 lb/day of silicon (from D4 and D5) entering the WWTF, 1.1 pounds of silicon was recovered from the boiler, equating to 8.9% by weight. These amounts are adjusted to account for 1 lb/day of siloxane lost through volatilization in the collection system and the additional 3 lb/day from residential sources. The results indicate the slug load that occurred on the sampling day was approximately 10 times higher than the average concentration for that time period.

Average Siloxane Flow

Methodology

The concentration of siloxane in the manufacturer's wastewater discharge was determined over a 3 year period before they closed to help establish the average siloxane loadings historically experienced by the WWTF. The concentrations in the wastewater were determined using a screening technique (a description of the analytical procedure can be found in the Appendix). The concentration of siloxane in the biogas was determined during this time period by analyzing samples according to Method 71 (a description of this method can be found in the Appendix).

Findings

NBC determined the concentrations in wastewater using a screening analysis as described in the previous section:

Siloxane from Manufacturer			
Sample Date	Manufacturer Flow (GPD)	Siloxane Concentration (ug/l)	Siloxane Flow (lb/day)
10/05/05	88,450	18,089	13.351
10/06/05	81,000	13,291	8.983
11/16/05	86,710	-1,725	-1.248
03/20/06	97,980	30,454	24.90
03/21/06	100,490	25,229	21.16
03/22/06	81,000	18,443	12.466
03/23/06	105,000	24,679	21.62
03/24/06	81,000	35,239	23.82
05/22/06	73,990	31,092	19.20
05/23/06	68,210	26,540	15.11
05/24/06	70,500	32,168	18.92
05/25/06	66,450	33,579	18.62
05/26/06	81,990	29,906	20.46
01/22/07	78,280	33,469	21.86
01/23/07	105,940	34,954	30.90
01/24/07	87,260	30,403	22.14
03/19/07	70,190	52,054	30
03/20/07	90,560	42,764	32.3
03/21/07	91,370	24,041	18.33
03/22/07	112,960	37,084	35.0
03/23/07	47,760	49,841	20
06/04/07	72,900	40,712	24.8
06/05/07	87,480	44,461	32.5
06/06/07	113,410	46,611	44
06/07/07	118,730	39,221	38.9
12/03/07	67,940	20,613	11.686
12/04/07	65,000	22,061	11.966
12/05/07	79,490	9,680	6.421
12/06/07	84,060	27,505	19.29
12/07/07	231,520	15,465	29.878
09/22/08	37,450	30,552	9.55
09/23/08	37,440	33,659	10.52
09/24/08	35,100	38,576	11.30
09/25/08	32,730	21,689	5.924
Average	83,245	29,776	20.1

Figure 10. Average Siloxane from Manufacturer during 3 Year Time Period

The results indicate the manufacturer discharged an average of 20.1 lb/day of siloxane during the period. It should be noted that the data set includes some data that was collected during times when slug loading was known to occur (3/19/07 through 6/7/07).

The amount of siloxane that entered the biogas during the same approximate time period was determined by collecting and analyzing the biogas according to Method 71 (Saeed, 2002):

Date	Siloxanes (ppmv)	Average Biogas (SCFH)	Siloxane (lb/day)
8/17/05	37.6	10,564	9.4
12/5/07	16.8	6,764	2.7
12/20/07	10.0	6,764	1.6
1/25/08	12.4	12,471	3.7
6/20/08	53.6	12,471	16
8/6/08	61.5	9,139	13
Average			7.8

Figure 11. Siloxane in Biogas during 3 Year Time Period

The results indicate that 7.8 lb/day of siloxane entered the biogas that was produced by the digester during this time period.

Findings

The potential amount of silica-based deposit that entered the boiler can be calculated based on biogas concentrations and flow rates. The average concentration of silica measured in the biogas during this time period was 229 ppmv and 55% of the biogas produced or 6,278 SCFH was burned in the boilers. It is known that burning the biogas can form a deposit that is 75.5% SiO_x (Tower, 2003). The potential amount of silica deposit can be calculated as follows:

$$\text{Deposit} = C \times 0.55 \times Q_{\text{tot}}$$

Equation 5

The results indicate that an average of 5.5 lb/day of silica entered the boiler.

The amount of deposit that was actually removed from the boilers was weighed every time the boilers were cleaned. Approximately 24.5% of this amount was comprised of non-silicon based soot. As the data shows in the following figure, an average of 2.6 lb/day of total deposit was removed from the boiler:

Silica Removed From Boilers	
Month	Silica Recovered (lb/day)
Oct-06	1.44
Nov-06	1.33
Dec-06	1.24
Jan-07	1.72
Feb-07	1.39
Mar-07	3.37
Apr-07	1.92
May-07	2.77
Jun-07	3.58
Jul-07	3.37
Aug-07	3.12
Sep-07	4.30
Oct-07	4.07
Dec-07	1.00
Jan-08	1.31
Feb-08	1.73
Mar-08	3.83
Apr-08	1.45
May-08	2.16
Jun-08	1.84
Jul-08	1.84
Aug-08	2.41
Sep-08	3.62
Oct-08	3.67
Average (silica)	2.6

Figure 12. Silica Removed from Boilers during 3 Year Time Period

The following figure summarizes the all the concentrations, flow rates and loadings that were determined during the 2 year time period when data was collected and the manufacturer was open:

Historic Average Values								
Waste Stream Location	Time Period		Concentration		Flow Rate		lb/day	WWTF _{inf} as Silicon ⁵
Manufacturer (3 year average siloxane) ¹	10/25/05	09/25/08	115	mg/l	83,245	GPD	20.1	8.30
Digester Biogas (3 year average siloxane) ²	08/17/05	08/06/08	32.0	ppmv	11,371	SCFH	7.78	2.95
Boiler (potential silica) ³	08/17/05	08/06/08	229	ppmv	6,278	SCFH	5.53	2.58
Boiler (deposit recovered) ⁴	10/05/06	10/29/08	75.5	%	-	-	2.64	0.93

Note¹: Concentration is from screening of manufacturer's discharge by NBC for D4 and D5

Note²: Concentration is from analysis of biogas by ESS laboratory for D3, D4, D5 and D6

Note³: Total silica concentration is from analysis of the biogas by ESS Laboratory

Note⁴: Deposit weight measured by NBC

Note⁵: Potential silica includes small amount from unidentified volatile silicon compounds in biogas

Figure 13. Historic Average Values

The results indicate that of the 8.30 lb/day of silicon (from D4 and D5) entering the WWTF, 2.95 lb/day entered the biogas, equating to 36% by weight. The results also indicate that 0.93 lb/day of deposit (as silicon) was recovered from the boiler, equating to 11% of the amount entering the WWTF. It should be noted that less biogas was burned during slug loading because the resulting lower boiler efficiency caused more natural gas to be burned instead.

Normal Siloxane Flow

A baseline or "normal" amount of siloxane in typical WWTF influent most commonly originates from ubiquitous residential sources. Variations above this amount are most likely due to industrial sources. A Swedish survey of WWTFs (Kaj, 2004) showed that the average concentration of siloxane in the influent of several WWTFs that were surveyed was 15 ug/l.

A Nordic survey of WWTFs (Kaj, 2005) indicates that the average siloxane concentration in anaerobic digested sludge is 27,000 ng/g on a dry weight basis. The concentration of siloxane in the wastewater entering the BP WWTF has not been measured since the manufacturer closed. Assuming the influent contains 15 ug/l and approximately 40% of the siloxane is released in the digester, then the BP digested sludge would theoretically contain 27,000 ng/g on a dry basis. The detectable limit for siloxanes in wastewater in this study was 10 ug/l.

Methodology

The concentration of siloxane in the biogas was measured several months after the manufacturer closed according to Method 71 and was found to be 2.43 ppmv. This can be compared to a total siloxane concentration of 0.1 to 5.0 ppmv found in biogas and landfill gas at other facilities (Arnold 2010) with landfills having lower concentrations than WWTFs. The biogas sample was also analyzed for total silica and was found to contain 24.3 ppmv.

Findings

The following figure summarizes all of the concentrations, flow rates and loadings that were determined during the most recent (current normal) time period after the manufacturer closed:

Current Normal Values								
Waste Stream Location	Time Period		Concentration		Flow Rate		lb/day	WWTF _{inf} as Silicon ⁵
WWTF Influent (FP siloxane) ¹	5/3/09	6/23/10	15	ug/l	23,640,004	GPD	2.96	1.12
Digester Biogas (siloxane) ²	5/29/09 10:20	5/29/09 13:20	2.43	ppmv	8,705	SCFH	0.503	0.190
Boiler (potential silica) ³	5/29/09 10:20	5/29/09 13:20	24.3	ppmv	4,522	SCFH	0.423	0.198
Boiler (deposit recovered) ⁴	5/21/09	6/23/10	75.5	%	-	-	0.185	0.065

Note¹: Concentration is estimated based on other WWTFs

Note²: Concentration is from analysis of biogas by ESS laboratory for D3, D4, D5 and D6

Note³: Total silica concentration is from analysis of the biogas by ESS Laboratory

Note⁴: Deposit weight measured by NBC

Note⁵: Potential silica includes small amount from unidentified volatile silicon compounds in biogas

Figure 14. Current Normal Values

The results indicate that of the 1.12 lb/day of silicon estimated to enter the WWTF, 0.19 lb/day entered the biogas, equating to 17% by weight. The results also indicate that 0.065 lb/day of the silicon was recovered from the boiler as a deposit, equating to 5.8% of the amount entering the WWTF.

Findings

The following figure shows the siloxane results from all 3 time periods:

Siloxane Volatization				
Time Period	Primary Clarifier TSS Removal	Digester Detention Time (days)	Siloxane Transferred into Biogas	
			(% entering WWTF)	(% entering Digester)
Maximum	47%	18	-	-
Historic Average	73%	18	36%	51%
Current Normal	54%	26	17%	32%

Figure 15. Siloxane Volatization

The results indicate that 32% to 51% of the siloxane entering the digester was released into the biogas during 2 different time periods. Approximately 50% of the volatile solids were destroyed

in the digester during both time periods. A significant amount of siloxane is not expected to biologically degrade (Grumpling, 1999). The amount of siloxane released from the solids is expected to approach this value since as the VSR (dX_{vss}) varies, the amount of siloxane released (dC_t) changes by a constant proportion according to the following differential form of the sorption equation:

$$\text{Siloxane Transferred into Biogas} = dC_t / dX_{vss} = K_p \times C_s$$

Equation 6

This simplified equation assumes C_t is much higher than C_s but not over-saturated, neglects the effect of mass transfer on C_s and does not take into account the effect of the high digester temperature that likely drives some of the absorbed siloxane off the solids. The result from the current normal time period suggests that resistance to mass transfer may be significant at the lower siloxane concentration. It indicates that when released by digestion, a significant amount of the initially adsorbed siloxane may redistribute onto remaining solids in the digester rather than release to the biogas. It is beyond the scope of this study to determine the effect of the liquid phase mass transfer resistance on the amount of siloxane transferred into the biogas.

The following figure shows the silica results from the 3 time periods:

Silica Deposition			
Time Period	Biogas Flared (%)	Silica Recovered from Boiler	
		(% of silicon entering WWTF)	(% of silicon entering Boiler) ¹
Maximum	54%	12%	35%
Historic Average	45%	15%	36%
Current Normal	48%	8%	33%

Note¹: Maximum time period value is estimated

Figure 16. Silica Deposition

The results indicate that 33% to 36% of the potential silicon-based deposit entering the boiler was recovered when the boiler was cleaned during the time period shown. It is estimated that 35% of the silicon deposited when loading was the highest, representing the highest actual amount recovered from the boiler. The value is extrapolated based on the other known values. This assumes that all of the siloxane was oxidized to silica in the boiler.

The results indicate that most of the silicon passed through the boiler. It is thought to pass through as a solid based on silica's melting point of 3,110 F, the average flame temperature of 2,600 F and a solid silica vapor pressure that is assumed to be insignificant. The amount of unburned siloxane and or gaseous silicon combustion byproducts is assumed to be insignificant.

The results indicate that the deposition, expressed as a percentage of the potential incoming silicon, was relatively constant. The siloxane loading varied greatly during the

different time periods as did the average boiler cycle time. The average cycle time varied from 6 days when the manufacturer was open to 45 days after they closed.

It is likely that the silicone dioxide particles deposited on the relatively cooler boiler tubes by a thermophoretic process where transfer was down the temperature gradient. During the boiler cycle, the effective boiler tube wall temperature increases as the insulating deposit accumulates and this decreases the rate of deposition with time. The rate of thermophoretic deposition of diesel soot has been observed to increase by a factor of 4 when an electrical current is applied (Cruz, 2009).

The Impact of Silica

Impact on Temperature

Siloxanes are known to oxidize to silicon dioxide (silica) when combusted and to form deposits that can impede heat transfer. At the Bucklin Point facility, high siloxane loadings shortened the biogas-fired sludge boiler cycle, increased stack gas temperature and increased fuel consumption. A diagram of the anaerobic digester heating system is shown in the following diagram:

Figure 17. Bucklin Point Digester Heat Balance

This study proposes that the thickness of the silica deposit that formed on the heat transfer surface reduced the overall heat transfer coefficient in the boiler. This reduced the amount of heat transferred into the hot water per unit of fuel burned which increased the temperature of the combustion gas that passed out the exhaust stack.

Methodology

The boiler was routinely cleaned and inspected. The thickness of the deposit was measured and found to be uniform over the length of the tubes. It is most likely that the entire amount of siloxanes entering the boiler was completely combusted and formed silica particles. The boilers were installed in 2004 and use 20% excess air. The 2,600°F flame temperature never exceeded the 3,110°F melting point of silica. The true density of the deposit was measured to be 2.3 g/ml which is nearly the density of silica. The bulk density of the material was measured to be 1.45 lb/ft³. A microscopic analysis by Liberty Mutual Laboratory (certificate of analysis can be found in the Appendix) determined it consisted of amorphous silica particles smaller than 5 microns. The conductivity of the deposit was measured by EMSL Laboratory and found to be 0.05 W/m2 at 300C (COA can be found in the Appendix).

Silica that accumulated on the stack gas thermometer probe mounted inside the boiler caused a negative bias in the stack gas temperature measurement. The bias was caused when the deposit insulated the probe from the hot stack gas. The bias was quantified by checking the thermometer measurements before and after cleaning the probe:

Deposit Impact on Temperature Measurement (F)				
Date	Average Deposit Thickness (mm)	Temperature before cleaning (F)	Temperature after cleaning (F)	Temperature Bias (F/mm)
7/14/08	1.7	570	621	-30.9
9/5/08	1.7	580	630	-30.0
Average Bias Correction Factor				30.5

Figure 18. Deposit Impact on Stack Gas Temperature Measurement

Findings

The stack gas temperature measurements used in this study were corrected by applying the above factor based on the measured thickness of the deposit. The following figure shows the corrected and uncorrected stack gas temperature measurements during 2009 when the biogas contained 24.3 ppmv of silicon:

Figure 19. Corrected and Uncorrected Stack Gas Temperature Measurement during 2009

The original uncorrected plot shows the temperature erroneously decreasing. The corrected plot shows the temperature properly increasing slowly as expected due to the relatively small amount of silica that deposited on the tubes and increased the heat loss.

The same correction factor was applied to the stack gas temperature measurements during June 2008 when the concentration of silicon in the biogas was 383 ppmv:

Figure 20. Corrected and Uncorrected Stack Gas Temperature Measurement during 2008

The data shows that the temperature increased quickly at first and then nearly leveled off later in the shortened boiler cycle. A steady silica deposition rate during the boiler cycle did not account for the large early temperature increase so silica was assumed to deposit as early as possible in the boiler cycle.

Impact on Boiler Efficiency

Over time, any boiler can accumulate soot from incomplete combustion that will decrease heat transfer, increase stack gas temperature and increase fuel consumption. A carbon soot layer 1/8 inch thick is known to reduce heat transfer by 8.5% (NCDENR 2004). However, silica should have a greater impact because it is 10 times more resistant to heat transfer than carbon soot based on published values of thermal conductivity.

The boilers at Bucklin Point fouled very quickly when the manufacturer discharged siloxane into the wastewater collection system. A similar phenomenon reportedly occurred at a WWTF located in Barrie, Canada. Their boiler efficiency reportedly dropped to as low as 20% during high siloxane loading according to OSB Laboratories of Canada. OSB developed the analytical

procedures used to measure the siloxane concentrations at the WWTF. There have been reports of silica deposits as much as ½ to 1 inch thick at other facilities.

Methodology

Bucklin Point collected information including stack gas temperatures, weights of silica produced and fuel consumption during every shift to help quantify siloxane's impact on the boilers. This data was used with information archived by the facilities SCADA system to determine the boiler's heat input, heat output and the heat transfer rate.

The potential amount of heat in the fuel input to the boiler can be determined from the following equation:

$$\text{Boiler Input} = h \times Q$$

Equation 7

where the Boiler Input is in units of BTU per hour, h is the measured heat value of the fuel input which was measured to be 547 BTU (Low Heat Value) per standard cubic foot of biogas and Q is the fuel flow rate in units of standard cubic feet per hour. A heat value of 1,000 Btu per standard cubic foot was used when the boiler was fed natural gas.

The heat output from the boiler was calculated based on the following equations:

$$\text{Boiler Output} = \Delta H_{\text{sludge}} / HTE_{\text{sludge}}$$

Equation 8

$$\Delta H_{\text{sludge}} = (Q \times \rho \times C_p)_{\text{sludge}} \times (T_{\text{out}} - T_{\text{in}})$$

Equation 9

where H is the amount of heat transferred to the sludge in units of BTU per hour, Q is the sludge flow rate in units of gallons per hour, ρ is the sludge density in units of pounds per gallon, C_p is the specific heat of the sludge in units of Btu per pound per °F and ΔT is the sludge temperature rise across the heat exchanger in °F. The sludge temperature measurements are archived by the facility's SCADA system. The density and specific heat are estimated to be that of water. HTE_{sludge} is the heat transfer efficiency of the sludge heat exchanger which was estimated to be 95%.

The boiler efficiency is defined as the difference between the boiler heat input and heat output divided by the heat input:

$$\text{Boiler Efficiency} = (\text{Input} - \text{Output}) / \text{Input}$$

Equation 10

Findings

The efficiency was determined during June 2008 when the manufacturer was open and the biogas contained 383 ppmv silicon and during June 2009 when manufacturer was closed and the biogas contained 24.3 ppmv silicon:

Figure 21. Boiler Efficiency during 2008 and 2009

The difference between the 2 plots is that the manufacturer discharged siloxanes during 2008 but not during 2009, illustrating the impact of high siloxane loading on the boiler efficiency. Heavy siloxane loading caused a 3.3 mm thick deposit that reduced the boiler efficiency to as low as 30% to 40%.

Impact on Heat Transfer

Methodology

To calculate the heat transfer rate in the boiler, the flame temperature, deposit thickness and overall heat transfer coefficient were determined. The flame temperature was calculated from the following equation based on the average specific heat of the combustion gasses and the known heat of combustion:

$$H_{\text{combustion}} = m \times C_p \times (T_{\text{flame}} - T_{\text{in}})$$

Equation 11

The rate of heat transfer in the boiler was calculated based on the sum of resistances to heat transfer according to the following equation:

$$\Delta H_{\text{boiler}} = A \times U \times \Delta T$$

Equation 12

$$\text{where } U = 1 / \sum R$$

Equation 13

Where A is the boiler heat transfer area in square feet, ΔT is the flame temperature minus the average stack gas temperature in °F. The gas side resistance and the resistance of the silica layer are the major components of the sum of resistance to heat transfer. In the absence of silica, the predominant contribution to the overall heat transfer coefficient is the inside gas heat transfer coefficient defined by (Gangapathy 2009):

$$h_i = 0.023 \times \text{Re}^{0.8} \times \text{Pr}^{0.4} \times 12 \times k / D$$

Equation 14

where k is the average thermal conductivity of the combustion gases and D is the inside diameter of the boiler tubes in inches. The Reynolds number is defined by:

$$\text{Re} = 15.2 \times w / D / \mu$$

Equation 15

where w is the mass flow rate through each boiler tube, D is the tube diameter and μ is the average viscosity of the combustion gases.

The Prandtl number is defined by the following equation:

$$\text{Pr} = \mu \times C_p / k$$

Equation 16

where C_p is the average specific heat of the combustion gasses.

The resistance of the silica layer is based its thermal conductivity that was measured by EMSL Laboratory (see appendix) and the thickness of the deposited layer. The thickness was calculated based on the amount of siloxane that entered the boiler and deposited.

Findings

The following figure contains the results and values used to calculate the boiler efficiency and heat transfer during June 2008:

Cummulative Volume Biogas Burned (SCF)	Fuel Input (MMBtu /hr) _{avg}	Boiler Output (MMBtu /hr) _{avg}	Combustioun Gas (lb/hr) _{avg}	Combustioun Gas (lb/hr) _{inst}	Stack Gas Temperature (F) _{meas}	Flame Temperature (F) _{calc}	Deposit Thickness (mm) _{avg}	Re	Heat Transfer Coefficient (W/m ² C)	Boiler Heat Transfer (MMBtu /hr) _{avg}
446	-	-	4,690	4,834	380	2,965	0.013	8,249	31.5	2.84
46,748	4.86	2.66	4,907	4,907	477	2,705	1.44	8,694	31.8	2.41
87,610	4.56	1.93	4,601	4,592	563	2,720	2.74	8,028	30.7	1.82
105,756	1.34	0.35	1,359	2,500	595	2,695	3.34	4,377	19.0	0.66
117,339	1.10	0.25	1,038	4,037	605	2,580	3.34	7,223	27.7	0.36
267,610	5.33	1.97	5,603	5,603	620	2,582	3.34	10,222	37.6	1.59
319,362	3.73	1.09	3,927	5,161	625	2,582	3.34	9,482	35.7	1.19
393,951	4.95	1.30	5,203	6,050	632	2,583	3.34	11,228	41.5	1.43
467,921	5.06	1.69	5,321	6,385	640	2,585	3.34	11,973	44.3	1.43
525,123	3.99	1.47	4,193	4,193	645	2,585	3.34	7,929	32.2	1.53
609,711	5.86	2.02	6,161	6,161	654	2,586	3.34	11,798	45.0	1.74
639,719	1.95	0.79	2,052	2,052	654	2,586	3.34	3,949	18.8	1.19

Figure 22. Values used to Determine Boiler Heat Transfer during June 2008

The amount of heat transferred based on the overall heat transfer coefficient ($f[U]$) was calculated under different conditions during 2008 to determine if the silica was responsible for the observed decrease in boiler efficiency. The heat transfer coefficient was calculated based on silica that deposited as quickly as possible and based on silica that deposited steadily during the boiler cycle:

Figure 23. Boiler Heat Transfer as a Function of Deposition

It can be seen that the quick depositing silica plot closely matches the calculated heat transfer rate determined from the sludge temperature rise whereas the steady deposit curve does not. It can be seen in the following graph that, upon closer inspection of the individual resistances to heart transfer, the overall resistance increased early in the boiler cycle and remained high throughout the boiler cycle due to the accumulation of silica on the boiler tubes:

Figure 24. Contributions to Boiler Overall Heat Transfer Resistance

The bar graph shows that the deposit reduced the boiler efficiency by approximately doubling the resistance to heat transfer. The graph shows that the resistance of the inside gas increased early in the cycle. This is when the average combustion gas temperature dropped (decreasing U and increasing R) and the deposit accumulated (increasing velocity and U and decreasing R). It can be seen that the temperature drop had the greater impact since R increased early in the cycle. The resistance decreased later in the cycle as the average combustion gas temperature increased.

Siloxane Condensation

Water is commonly condensed from biogas as the fuel is conditioned prior to a reciprocating engine or activated carbon. Water-insoluble siloxanes may also condense at the same time depending on the type of siloxane present, the initial concentration and the temperature. It has been approximated that when the concentration of siloxane in the BP biogas was high, approximately half could be removed at 40°F and 90% can be removed at -10°F. This study proposes that the amount of siloxane removed can be predicted using available thermodynamic data and chemical engineering theory.

The biogas from the Bucklin Point WWTF contained unusually high concentrations of D4 and D5 siloxane that originated from a manufacturer of personal care products. The average biogas composition and characteristics are listed in the following figure:

Bioags Content	
Parameter	Average
Methane (% by volume)	61.8%
Carbon Dioxide (% by volume)	36.4%
Oxygen (% by volume)	0.25%
Nitrogen (% by volume)	1.0%
Water (saturated at biogas temperature) ₁	3.90%
Hydrogen Sulfide (ppmv) ₂	149
Chlorinated VOCs (ppmv) _{max}	7.27
Non-Chlorinated VOCs (ppmv) _{max}	34.6
Siloxanes (ppmv) _{avg}	32.0
Biogas Temperature (°F entering boiler) ₃	86.0
Heat Value (HHV for dry gas in Btu/cf)	616

Note 1: Estimated water content in biogas is a calculated based on temperature

Note 2: 2009 hydrogen sulfide concentration based on samples collected 2/29/08 to 6/2/09

Note 3: Average biogas temperature are based on measurements from 2008

Figure 25. Biogas Content and Characteristics

The water vapor and siloxanes in the biogas may condense together forming a liquid condensate containing an aqueous phase and a separate siloxane phase. It is also possible that D4 may deposit as a solid due to its high freezing point of 64°F.

The theoretical amount of D4 and D5 in the biogas can be calculated based on Raoult's Law that is expressed in the following equation for a non-ideal binary mixture:

$$\sum (\phi_i \times Y_i \times P_T) = \sum (X_i \times P_i^*(T))$$

Equation 17

where ϕ_i is the fugacity coefficient of the component in the gas phase, Y_i is the gas phase mole fraction, P_T is the total pressure, X_i is the liquid phase mole fraction and $P_i^*(T)$ is the saturation vapor pressure of the pure component at temperature (T). The activity coefficient of the D5 can be estimated to be unity since it can be considered the solvent in the condensate. The activity coefficient of the liquid D4 solute may be near unity based on its similarity to D5. However, if D4 deposits as a solid then this would affect the D4 mole fraction in the liquid phase.

The saturation vapor pressure of pure D4 and D5 siloxanes can be calculated based on published Antoine constants (Palczewska-Tulinska 2005) that were defined over a temperature range of 101.57 to 343.60 °F and 167.14 to 403.48 °F respectively:

$$\ln(P_{D4}^*) = 13.47185 - 2906.90/(T - 79.496)$$

Equation 18

$$\ln(P_{D5}^*) = 12.67409 - 2772.84/(T - 135.502)$$

Equation 18

where P_i^* is the saturation vapor pressure of the pure component in kPa and T is the temperature of the vapor phase in °K.

Methodology

The biogas from Bucklin Point was passed through a pilot chiller as shown in the following diagram where it was cooled to approximately 34°F:

Figure 26. Pilot Chiller

The raw biogas was chilled by passing it at an average flow rate of 160 ml/min through a 1/8 inch diameter x 25 foot long column immersed in ice (see Appendix for pilot chiller

specifications). The inlet and outlet concentrations of D4 and D5 siloxanes were measured in samples that were collected using Method 71 and analyzed by ESS Laboratory using GM/MS technique.

The predicted outlet concentrations were calculated according to the previously defined equations to determine if actual removals agreed with theory. The initial temperature of the biogas was 94.5°F and the initial concentrations of D4 and D5 were 9.3 ppmv and 52.2 ppmv respectively. Binary thermodynamic theory predicts D4 and D5 will be reduced to 5.4 and 5.3 ppmv respectively in the chilled biogas. This assumes that Antoine constants are valid at 34°F which is below the temperature range in which they were defined. It also assumes that the liquid phase activity coefficients are 1. Very little data has been published on the thermodynamic properties of D4 and D5 and there is no published data on the behavior of a mixture of D4 and D5. Upon start-up, the chilled biogas quickly reached the average operating temperature of approximately 33.9°F (see appendix for operating data). The chilled biogas temperature was measured in a glass condensate trap at the chiller outlet as shown in the following pictures:

Figure 27. Pilot Chiller and Condensate Trap

Findings

The following concentrations of siloxanes in the biogas were determined before and after chilling and are compared to the concentrations predicted by theory in the following graphs. The theoretical concentrations were determined by assuming ideal binary component behavior (activity coefficients =1):

Figure 28. Pilot Chiller Results

The results show that the biogas leaving the pilot chiller contained 7.22 and 12.6 ppmv of D4 and D5 siloxanes respectively. These concentrations were higher than the 5.49 and 5.34 ppmv predicted by theory. The initial and final partial pressures are summarized in the following figure:

8/6/07 Siloxane Partial Pressures x 10 ⁶ (atm)				
Temperature (F)	Actual D4 Partial Pressure	Pure D4 Saturation Pressure	Actual D5 Partial Pressure	Pure D5 Saturation Pressure
94.5	9.33	2,520	52.2	325
33.9	7.22	122	12.6	6.56
$X_i =$	0.05		0.95	
P_i (ideal) =	6.17		6.23	
$\phi_i =$	0.85		0.49	
Predicted Removal		34%		88%
Actual Removal		23%		76%

Figure 29. Comparison of Actual and Theoretical Siloxane Partial Pressure

Findings

The values of ϕ shown in Figure 29 are the ideal partial pressure at 34°F divided by the measured partial pressure. These values would represent the fugacity coefficient of D4 and D5 if the ideal predicted partial pressure was known to be valid at the temperature, if the experimental condensate trap was known to be 100% efficient at stopping carryover of siloxane particulates out of the pilot chiller and if there were no other compounds present in the condensate.

The results show that D4 and D5 siloxane removals can be approximated using available information. However, the overall predicted removal was 12% greater than the actual removal measured in the experimental chiller operated at 34°F.

The ideal partial pressures are based on Antoine constants that were defined in a higher temperatures range. In addition, the Antoine constants for the D4 liquid/vapor were used at 34°F which is below the 64°F freezing point of pure D4. It is possible that cooling the unsaturated biogas caused solid D4 to deposit with liquid D5. The Antoine constants were used in this temperature range because more accurate data is not available. D4 and D5 siloxane vapor pressure data has also been published (Colona 2006). However, it too is defined well above the temperatures in this study.

The calculations used an overall D4 mole fraction in the condensate. In effect, this assumes that any D4 solid that may have formed has the same pure component vapor pressure as its liquid counterpart. The activity of any solid D4 is unity and the activity of the liquid D4 is unknown but assumed to be unity. All of these assumptions and simplifications very likely introduced more error into the calculation than was found in the data set published by Colona (3% maximum error).

The results of this study indicate that siloxane removal can be predicted, but the resulting error is substantial and cannot be attributed to a single factor such inaccurate Antoine constants, non-ideality or possible experimental pass-through of siloxane through the condensate trap.

Experimental error is not likely a predominant contributing factor since repeat runs yielded similar D5 results with a standard deviation of 5.3%.

The accuracy of making such predictions of siloxane removal in a chiller could likely be improved by using Antoine constants that are defined in temperature range of interest. Additionally, determining the vapor pressure, concentration and activity of solid and liquid components separately would likely improve the accuracy of the prediction.

CHAPTER 4

CONCLUSIONS

The portion of siloxane that was released into the biogas was the same as the portion of volatile solids that were digested at a high siloxane concentration. A smaller portion was released when the siloxane concentration was low. This indicates that resistance to mass transfer likely plays a role in how much is released as well as the amount of volatile solids digested at a low siloxane concentration.

At a high siloxane concentration, silica deposited quickly deposited in the boiler and impacted the overall heat transfer coefficient as expected based primarily on the low conductivity of the deposit.

The partial pressure of D4 and D5 siloxanes were reduced by condensation in a pilot biogas chiller at 34F. Actual removals were 12% less than the removal predicted by theory.

APPENDIX 1 – PARTITION COEFFICIENTS

Theoretical Partition Coefficients

Equilibrium Siloxane Concentrations	Phase	K_p (L/g)	Aq. (mg/l) Slidg (ug/l)
Primary Sludge	Aqueous D4	36	0.056
	Sludge D4		66,780
	Aqueous D5	89	0.017
Waste Activated Sludge	Sludge D5		50,118
	Aqueous D4	8	0.056
	Sludge D4		14,840
	Aqueous D5	16	0.017
	Sludge D5		9,010

VSS = 33,125 mg/l = 3.3%
 Digester Sludge Composition 50% D5 53
 Digester Sludge Composition 50% D4 22

Digester Influent Sludge Partition Coefficients

Equilibrium Siloxane Concentrations	Phase	K_p (L/g)	Aq. (mg/l) Slidg (ug/l)
Digester Sludge	Aqueous D4	22	0
	Sludge D4		0
	Aqueous D5	29	0.079
	Sludge D5		75,000

Digester Influent D5 Partition Coefficient

Sample Matrix	Concentration (mg/l)	K_p (L/g)
Aqueous	0.079	29
Total	75

APPENDIX 2 – SILICA REMOVED FROM BOILERS

Silica Removed From Boilers

Boiler #	Date	Interval (days)	Silica Collected (lb)	Daily Rate (lb/day)	Month	Average Monthly Rate (lb/day)
1	9/28/06		9.50		Dec-00	
1	10/5/06	7.0	5.25	0.75	Oct-06	1.44
3	10/12/06	7.0	14.50	2.07	Nov-06	1.33
1	10/18/06	6.0	9.25	1.54	Dec-06	1.24
3	10/25/06	7.0	9.75	1.39	Jan-07	1.72
1	11/1/06	7.0	9.50	1.36	Feb-07	1.39
3	11/9/06	8.0	7.25	0.91	Mar-07	3.37
3	11/16/06	7.0	14.00	2.00	Apr-07	1.92
1	11/22/06	6.0	7.50	1.25	May-07	2.77
3	11/30/06	8.0	9.00	1.13	Jun-07	3.58
2	12/7/06	7.0	8.50	1.21	Jul-07	3.37
3	12/13/06	6.0	8.50	1.42	Aug-07	3.12
1	12/21/06	8.0	6.00	0.75	Sep-07	4.30
2	12/28/06	7.0	11.00	1.57	Oct-07	4.07
3	1/3/07	6.0	11.50	1.92	Dec-07	1.00
1	1/10/07	7.0	11.75	1.68	Jan-08	1.31
2	1/17/07	7.0	13.25	1.89	Feb-08	1.73
1	1/25/07	8.0	11.25	1.41	Mar-08	3.83
2	2/1/07	7.0	8.25	1.18	Apr-08	1.45
3	2/6/07	5.0	6.75	1.35	May-08	2.16
2	2/15/07	9.0	10.50	1.17	Jun-08	1.84
1	2/21/07	6.0	10.00	1.67	Jul-08	1.84
3	2/28/07	7.0	11.25	1.61	Aug-08	2.41
2	3/5/07	5.0	14.00	2.80	Sep-08	3.62
1	3/7/07	2.0	8.50	4.25	Oct-08	3.67
3	3/14/07	7.0	8.50	1.21	Nov-08	1.90
2	3/27/07	13.0	16.00	1.23	Dec-08	0.23
3	3/29/07	2.0	14.75	7.38	Jan-09	0.49
1	4/3/07	5.0	7.25	1.45	Feb-09	0.42
2	4/12/07	9.0	14.25	1.58	Apr-09	0.28
3	4/19/07	7.0	22.50	3.21	May-09	0.21
1	4/27/07	8.0	11.50	1.44	Aug-09	0.12
2	5/2/07	5.0	13.75	2.75	Oct-09	0.28

Silica from Boilers (all points)

Silica from Boilers (monthly avg)

3	9/4/08	7.0	26.00	3.71
1	9/10/08	6.0	9.00	1.50
2	9/11/08	1.0	11.00	11.00
1	9/16/08	5.0	7.00	1.40
3	9/16/08	5.0	8.50	1.70
1	9/23/08	7.0	10.25	1.46
2	9/25/08	2.0	10.25	5.13
3	9/30/08	5.0	15.25	3.05
1	10/1/08	1.0	9.25	9.25
2	10/7/08	6.0	8.50	1.42
3	10/15/08	8.0	11.00	1.38
3	10/20/08	5.0	11.00	2.20
1	10/22/08	2.0	8.00	4.00
2	10/23/08	1.0	5.50	5.50
3	10/29/08	6.0	11.50	1.92
1	11/7/08	9.0	8.50	0.94
1	11/11/08	4.0	6.00	1.50
2	11/13/08	2.0	6.50	3.25
-	12/11/08	28.0	6.50	0.23
-	1/7/09	27.0	6.00	0.22
-	1/17/09	10.0	7.50	0.75
3	2/12/09	26.0	9.00	0.35
1	2/23/09	11.0	5.50	0.50
2	4/6/09	42.0	7.00	0.17
3	4/21/09	15.0	6.00	0.40
3	5/21/09	30.0	6.25	0.21
3	8/18/09	89.0	11.00	0.12
2	10/7/09	50.0	5.00	0.10
1	10/19/09	12.0	5.50	0.46
3	11/5/09	17.0	4.25	0.25
2	12/23/09	48.0	5.10	0.11
3	2/10/10	49.0	6.25	0.13
3	6/23/10	133.0	14.25	0.11

1	12/18/07	6.0	4.50	0.75
2	12/27/07	9.0	13.25	1.47
3	1/3/08	7.0	9.25	1.32
1	1/10/08	7.0	9.00	1.29
2	1/16/08	6.0	8.00	1.33
1	2/12/08	27.0	10.50	0.39
2	2/21/08	9.0	13.25	1.47
3	2/24/08	3.0	10.00	3.33
3	3/4/08	9.0	7.50	0.83
1	3/6/08	2.0	18.50	9.25
1	3/9/08	3.0	7.50	2.50
3	3/15/08	6.0	9.50	1.58
2	3/19/08	4.0	20.00	5.00
3	4/1/08	13.0	14.50	1.12
2	4/16/08	15.0	19.00	1.27
3	4/23/08	7.0	12.50	1.79
1	4/30/08	7.0	11.50	1.64
2	5/6/08	6.0	13.50	2.25
1	5/20/08	14.0	9.25	0.66
2	5/23/08	3.0	11.00	3.67
3	5/28/08	5.0	10.25	2.05
1	6/9/08	12.0	11.00	0.92
2	6/12/08	3.0	11.00	3.67
3	6/19/08	7.0	11.00	1.57
1	6/26/08	7.0	8.50	1.21
2	7/2/08	6.0	9.50	1.58
3	7/14/08	12.0	11.00	0.92
1	7/17/08	3.0	10.00	3.33
2	7/24/08	7.0	10.75	1.54
3	7/31/08	7.0	12.75	1.82
1	8/6/08	6.0	9.00	1.50
2	8/6/08	6.0	12.00	2.00
3	8/12/08	6.0	9.50	1.58
2	8/19/08	7.0	9.50	1.36
3	8/21/08	2.0	8.50	4.25
1	8/26/08	5.0	8.50	1.70
2	8/28/08	2.0	9.00	4.50

		Nov-09 Jan-09		0.25	
3	5/10/07	8.0	30.25	3.78	
2	5/17/07	7.0	16.50	2.36	
3	5/26/07	9.0	24.50	2.72	
1	5/30/07	4.0	9.00	2.25	
1	6/5/07	6.0	10.00	1.67	
2	6/5/07	6.0	18.00	3.00	
3	6/14/07	9.0	23.50	2.61	
1	6/18/07	4.0	9.00	2.25	
2	6/20/07	2.0	5.00	2.50	
3	6/26/07	6.0	18.00	3.00	
1	6/27/07	1.0	10.00	10.00	
1	7/2/07	5.0	11.25	2.25	
1	7/5/07	3.0	18.00	6.00	
1	7/10/07	5.0	9.25	1.85	
2	7/12/07	2.0	11.50	5.75	
3	7/19/07	7.0	20.00	2.86	
1	7/26/07	7.0	10.50	1.50	
2	8/2/07	7.0	9.00	1.29	
3	8/8/07	6.0	18.50	3.08	
1	8/21/07	13.0	11.25	0.87	
3	8/23/07	2.0	16.50	8.25	
2	8/30/07	7.0	14.75	2.11	
3	9/6/07	7.0	14.50	2.07	
1	9/12/07	6.0	9.25	1.54	
3	9/13/07	1.0	10.00	10.00	
2	9/19/07	6.0	9.00	1.50	
3	9/20/07	1.0	10.00	10.00	
1	9/27/07	7.0	4.75	0.68	
2	10/3/07	6.0	10.00	1.67	
3	10/4/07	1.0	8.00	8.00	
2	10/10/07	6.0	8.00	1.33	
2	10/11/07	1.0	9.50	9.50	
3	10/18/07	7.0	13.50	1.93	
1	10/24/07	6.0	6.50	1.08	
2	10/26/07	2.0	10.00	5.00	
2	12/6/07	15.0	9.00	0.60	
3	12/12/07	6.0	7.00	1.17	

APPENDIX 3 – SILICA DEPOSIT DENSITY

Silica Deposit Density (from NBC lab measurements 12/11/09):

Density	Volume (milliliters)	Mass (grams)	Density (g/ml)	Density (lb/ft ³)
Loose (12/11/09)	11	0.255	0.023	1.45
Tapped (12/11/09)	8.2	0.255	0.031	1.94
Packed (12/11/09)	2.85	0.255	0.089	5.58
True (water displacement) (1/1/08)	2.0	4.4	2.3	146

Density of fumed silica is 2.2 g/cm³ according to Wikipedia on 7/22/2010

APPENDIX 4 – BOILER HEAT TRANSFER RATE

Boiler Heat Transfer based on Overall Transfer Coefficient

Boiler Operating Conditions		Temperatures (F)		IN	OUT	AVG	DIFF
Inside Tube Diameter (inches)	2.75	Combustion Gas	2,586		654	1,620	2,386
Inner (gas) heat transfer coeff (W/m ² C)	18.8	Hot Water	165		200	183	489
Outer (liquid) heat transfer coeff (W/m ² C)	1,000	Log Mean Temperature Difference					1,197
Total heat transfer surface area (sq.ft.)	563	Temperature Correction Factor (.75 - .97)					0.970
Typical Natural Gas Flame Temperature (F)							3,542

Theoretical Heat Output Calculation

Resistance	Thickness (mm)	Inside Radius R _i (mm)	Thermal Conductivity (W/m°C)	Resistance	
				[C/(W/m)]	(%ofTot)
Inside Tube	-	0.0	-	0.053	55%
Silica	3.340	31.6	0.074	0.043	44%
Tube Wall	3.174	34.9	50	0.000	0%
Scale	0.000	38.1	1.21	0.000	0%
Outside Tube	-	38.1	-	0.00083	1%
Heat (W)	348,004	1.19 MMBtu/hr	0.0970	100%

Estimated Silca Conductivity at 884°C

Temp (K)	Temp (°C)	Conductivity (W/m°C)
373	100	0.049
573	300	0.056
884	789	0.074

APPENDIX 5 – EQUIPMENT SPECIFICATION

Siloxane Study Equipment Specifications:

WWT Facility Equipment – Flow Meters:

Influent (Dry Weather Primary Influent) Wastewater Flow Meter

Manufacturer: Krohne Holland Altometer

Model: IFC090

Options: LA S2/5

Type: Magnetic Flow Meter

Number: IFC090F/D/H/36 AO2 62313

Flow Range: 0 – 65 MG/DAY

GK: 5.831

I: 4 – 20 Ma

RI<=0.5KOhm

P: 1000 P/S

Tag: FIT-0303

Sludge (Primary) Pump Flow Meter No. 1

Manufacturer: Krohne Holland Altometer

Model: IFC090

Type: Magnetic Flow Meter

Number: IFC090/F/D/HART/6, 0249/03 ISOKL.HIP67

Flow Range: 0 – 1,000 Gal/min

GK: 3.2597

I: 4 – 20 Ma

RI<=0.5KOhm

P: 1000 P/S

DN 150 mm/6 inch – PUI-HC-PN150PSI

Sludge (Primary) Pump Flow Meter No. 2

Manufacturer: Krohne Holland Altometer

Model Number: IFC090/F/D/HART/6, 0280/03 ISOKL.HIP67

Flow Range: 0 – 1,000 Gal/min

GK: 3.1791

I: 4 – 20 Ma

RI<=0.5KOhm

P: 1000 P/S

DN 150 mm/6 inch – PUI-HC-PN150PSI

Sludge (Primary) Pump Flow Meter No. 3

Manufacturer: Krohne Holland Altometer

Model Number: IFC090/F/D/HART/6, 028/03 ISOKL.HIP67

Flow Range: 0 – 1,000 Gal/min

GK: 3.2265
I: 4 – 20 Ma
RI<=0.5KOhm
P: 1000 P/S
DN 150 mm/6 inch – PUI-HC-PN150PSI

DAF Sludge Flow Meter Nos. 1 & 2

Manufacturer: DanFoss, Stonehouse, GL102LU, UK
Type: Magnetic Flow Meter
Model: 3100 Mag Water
Type Number 141F211000
Code Number: 08328056
Serial Number: 182989T222, 117130T262
Calibration Factor: 17.74950, 17.74950
Size DN: 150
Year Manufactured: 2002
Flange Standard ASME B16.5, Class 150, Electrodes A1S1 316Ti, Neoprene Lining

Scum Flow Rate Meter

Manufacturer: Krohne Holland Altometer
Model Number: IFC090/F/D/HART/6, 0248/03 ISOKL.H IP67
Flow Range: 0 – 1,000 Gal/min
GK: 2.6279
I: 4 – 20 Ma
RI<=0.5KOhm
P: 1000 P/S
DN 100 mm/4 inch – PUI-HC-PN150PSI

Total Sludge Influent to Anaerobic Digester Flow Meter

Manufacturer: Siemens
Model: Sitrans FM Magflo MAG6000
Model Number: FDK: 083F5003
TAG: FIT 1050

Biogas Flow Meters (5)

Manufacturer: Sage Metering Inc., 8 Harris Court, DI, Monterey, CA 93940, 831-242-2030
Type: Insertion Type Constant Temperature Thermal Mass Flow Meter, with sensing element utilizing 2 RTDs, factory calibrated, and has 4 independent calibrations available (Channels A – D)
Accuracy: +/- 1% of Reading
Repeatability: 0.2% of Full Scale

Portable Meter

Manufacturer: Thermo Electron Corp.
Model: Polysonics HYDRA SX30

Type: Strap-On Ultrasonic

WWT Facility Equipment – Process Equipment:

Primary Digesters (3)

Type: Anaerobic Digester

Mixing: Complete mix

Mixers: three 10 hp impeller-type mixers per digester, each with 23" diameter draft tube

Cover: Fixed

Tank Side-Walls: 27.5 ft insulated

Liquid Depth: 26 feet sidewall depth

Tank Diameter: 75 feet

Temperature Set Point: 95 F to 97F

Heating is by 3 biogas fired sludge boilers and 3 heat exchangers

Biogas Output pressure: 9" W.C. typical

Secondary Digester (1)

Type: Sludge Holding Tank

Mixing: none

Temperature Set Point: none

Heating: none

Tank Sidewalls: 16 ft (approximate)

Cover: Floating Cover

Tank Diameter: 70 feet

Boilers (3)

Manufacturer: Burnham Commercial, Lancaster PA 17605

Model 4F Series #563 fire-tube wetback three-pass commercial steel boiler

Forced draft with modulating burners, Operable on natural gas or digester gas

Sales Order No. 691423

Model Number: 4FPW563A3-SPL-G-GP

National Boiler Number: 27960, 27961, 27962

Gas Input: 3891.3 MBH

Gross Output: 3113.0 MBH, 4720.0 MBH Water, 93 BHP

Firing Rate 4713 MBH, Net Water Rating 4106

Max Working Pressure 60 psi water, Heating Surface 563 Sq Ft (fireside)

Boiler Burners (3)

Manufacturer: Gordon Piatt

Model Number: R12.8-GG-30/030011, operable on natural gas or digester gas

MAX 3891 MBH, MIN 1297 MBH

Date Code 02/03

Serial Number: 030009, 030010,

Capacity: 1,297 MBH – 3,891 MBH Gas

Combustion Control: FIREYE E110, E340, E30

Mainfold Pressure 4.55/4.75" WC, 0.5" WC

Gas Input Supply Pressure: 24" (High Fire) – 27" (Low Fire)

Flue Gas Oxygen: 4.6% (High fire) – 5.9% (Low fire)

Heat Exchangers No. 1

Manufacturer: Walker Process Equipment , Aura, IL

Model Number: E-1500

Capacity: 1,500,000 Btu/hr

Serial Number: P 20760-1

Heat Exchangers No. 2

Manufacturer: Walker Process Equipment , Aura, IL

Model Number: E-1500

Capacity: 1,500,000 Btu/hr

Serial Number: P 20760-2

Heat Exchangers No. 3

Manufacturer: Walker Process Equipment , Aura, IL

Model Number: E-1500

Capacity: 1,500,000 Btu/hr

Serial Number: P 20760-3

Biogas Booster Pumps (2)

Manufacturer: Spencer Turbine Company, Windsor, CT

Model Number: GH-050g-H-MOD

Serial Number: 801950

Unit Space Heaters (3 in Digester Building)

Manufacturer: Sterling a Division of Mestek, Inc., Westfield, MA 01085

Type: hot water

Model Number: HS 84E

Unit Number: HSUA HSB0841206

Serial Number: P02347105006002

Output: 61,000 Btu/hr

Maximum Air Temperature: 200 F

CFM: 1,400

Test Pressure: 250

Electrical: 115 V, 1 Phase, 60 HZ, 3.0 amps, 1/8 hp, 3.8 MCA, Max Fuse 15

Unit Space Heaters (1 in Flare Control Building)

Manufacturer: Sterling a Division of Mestek, Inc., Westfield, MA 01085

Type: Hot Water

Model Number: HS 84E

Unit Number: HSUA HSB0841206

Serial Number: P02347105006002

Output: 61,000 Btu/hr

Maximum Air Temperature: 200 F

CFM: 1,400

Test Pressure: 250

Electrical: 115 V, 1 Phase, 60 HZ, 3.0 amps, 1/8 hp, 3.8 MCA, Max Fuse 15

Unit Space Heaters (7 in HEX Building)

Manufacturer: Sterling a Division of Mestek, Inc., Westfield, MA 01085

Type: Hot Water

Model Number: HS 84E

Unit Number: HSUA HSB0841206

Serial Number: P02347105006002

Output: 61,000 Btu/hr

Maximum Air Temperature: 200 F

CFM: 1,400

Test Pressure: 250

Electrical: 115 V, 1 Phase, 60 HZ, 3.0 amps, 1/8 hp, 3.8 MCA, Max Fuse 15

Tag: DG-UH-1 through 7

WWT Facility Equipment – Temperature Sensors:

Digester Sludge Temperature Sensors (6)

Manufacturer: Winters Instruments, Toronto, Ontario, Canada, M3A 1B2, (716) 874-8700

Type: RTD

Tag Number: Supply Sludge TE-1095-1, 2 and 3 and Return Sludge TE-1096-1, 2 and 3

Model Number: 12924/S43643

Part Number: R1T185L3-S4D0608-8HN71-SL

Type: Resistance temperature detector (RTD)

Inlet sludge temperature is TE1095-1, 2, and 3

Outlet (heated) sludge temperature is TE1096-1, 2 and 3

Biogas Temperature Sensors

Manufacturer: Winters Instruments, Toronto, Ontario, Canada, M3A 1B2, (716) 874-8700

Type: RTD

Tag Number: TE-1150-1 and TE-1150-2

Model Number: 12924/S43643

Part Number: R1T185L3-S4D0608-8HN71-SL

Stack Gas Thermometers (3)

Manufacturer: Tell-Tru Manufacturing Company, Rochester, NY

Type: Bimetal Insertion Thermometer

Dual Range: 150/750 F and 50/400 C with 3" dial

Model: GT-300

Stem: 0.375" dia. X 9" long, 7/8" NPT connection

Accuracy: +/- 1% of full span

Sampling, Analytical and Pilot Condenser Equipment and Instruments:

Condenser

25 foot coil of 1/8" diameter copper tubing, solvent cleaned, backed and purged with argon, packed in 15 gallons of ice, with 150 ml condensate trap with thermometer

Condenser Thermometer

Manufacturer: Orion

Type: Digital Pocket Thermometer

Range: -58F to 302F

Model: ERT 600

Serial Number: 103437

Biogas Sample Flow Meter

Manufacturer: Dwyer Instruments, Inc., Michigan City, IN

Model: RMA-12

Range: 0 – 500 ml/min

Accuracy: +/- 4% full scale reading

Biogas Sample Impingers (2)

Manufacturer: Wilmad-Labglass

Type: Graduated midget bubbler/impinger, extra coarse fritted dispersion sparger, with 24/40 ground glass stopper, reference R.I.3360 Bureau of Mines

Capacity: 30 ml maximum liquid capacity

Model: LG-6893

Silica Scale

Manufacturer: Berkley

Type: Analog Pocket Scale

Range: 0 to 28 pounds

Increments: 0.5 lb

Laboratory Centrifuge

Manufacturer: Eppendorf AG 22331, Hamburg, Germany

Model: 5804

Model Number: 5804 no. 0011322

Maximum Speed: 14,000 min⁻¹

Kinetic Energy: 27,450 Nm

Maximum Density: 1.2 kg/dm³

Year Manufactured: 2005

Power: 950 W, 120 V, 11 A, 50 – 60 Hz

Screening Gas Chromatograph

Clarus 500 with Tekmar Velocity XPT Sample Concentrator (purge & trap) and 60 meter x 0.25 mm ID (1.4 um df) Perkin Elmer Elite 624 column (see Analytical Method for complete

description). Temperature ramp: 35 C initial temperature, 3.00 minute initial hold time, first ramp is 7C/minute to 100 C and hold for 0 minutes, second ramp is 15C/minute to 225 C and hold for 3.5 minutes and final ramp is 15C/min to 225 C and hold for 5 minutes

APPENDIX 6 – BIOGAS SAMPLE VOLUME DETERMINATION

Time	Sample Flow (Hundred ml/min)	Sample Volume (Hundred ml)	Operating Data
0:00:00	1.30	5.5	Sample Start Time 1/25/08 12:00
0:04:00	1.45	1.4	Sample End Time 1/25/08 15:00
0:05:00	1.30	1.4	Biogas Temperature 74.3 F (pipeline)
0:06:00	1.50	1.6	Biogas Pressure 14.9 inches WC
0:07:00	1.75	1.5	Booster Pump Run Time 3 hours
0:08:00	1.35	10.3	Biogas Data
0:15:00	1.60	3.1	Specific Gravity 0.91
0:17:00	1.50	13.0	Specific Volume (STP) 22.3 liters/gmole
0:25:00	1.75	1.5	<p>Sample Flowrate</p> <p>Flowrate (hundred ml/min)</p> <p>Sampling Time (Hours:Minutes)</p>
0:26:00	1.25	3.4	
0:29:00	1.05	9.3	
0:36:00	1.60	1.6	
0:37:00	1.60	20.8	
0:50:00	1.60	6.2	
0:55:00	0.90	1.1	
0:56:00	1.25	19.2	
1:10:00	1.50	15.0	
1:20:00	1.50	7.5	
1:25:00	1.50	7.5	
1:30:00	1.50	7.5	
1:35:00	1.50	7.7	
1:40:00	1.60	16.0	
1:50:00	1.60	16.0	
2:00:00	1.60	7.7	
2:05:00	1.50	15.5	
2:15:00	1.60	15.5	
2:25:00	1.50	10.5	
2:32:00	1.50	7.5	
2:37:00	1.50	6.0	
2:41:00	1.51	7.5	
2:46:00	1.50	9.0	
2:52:00	1.51	6.0	
2:56:00	1.51	3.0	
2:58:00	1.51	3.0	
3:00:00	1.51		
Sum			27.0 liters (as air)
Average Flow Rate			150 ml/min
SpGr Correction Factor			1.048 dimensionless multiplier
Sample Temperature			4.0 Celcius
Total Sample Volume			27.9 liters of biogas @STP

Mass and Concentrations

Compound	MW	Front (ug)	Back (ug)	Carryover	Total (ppmv)
Silicon	28.09	4,123	67	2%	119.7
D4	296.62	226	0	0%	0.61
D5	370.77	5,394	66	1%	11.81

Silica/Siloxane Molar Ratio

1.95

Time	Chilled Flow (Hundred ml/min)	Chilled Volume (Hundred ml)	Sample Temp (F)	Operating Data
0:00:00	1.30	8.05	32.3	Sample Start Time 1/25/08 12:00
0:07:00	1.00	3.45	32.3	Sample End Time 1/25/08 15:00
0:10:00	1.30	1.35	32.3	Biogas Temperature 74.3 F (pipeline)
0:11:00	1.40	18.20	32.3	Biogas Pressure 14.9 inches WC
0:25:00	1.20	4.50	32.1	Booster Pump Run Time 3 hours
0:29:00	1.05	7.53		Biogas Data
0:36:00	1.10	1.13		Specific Gravity 0.91
0:37:00	1.15	15.27		Specific Volume (STP) 22.3 liters/gmole
0:50:00	1.20	5.00	32.0	<p>Sample Flowrate</p>
0:55:00	0.80	1.03	32.0	
0:56:00	1.25	20.65	32.0	
1:10:00	1.70	17.00	32.0	
1:20:00	1.70	8.25		
1:25:00	1.60	8.00	32.0	
1:30:00	1.60	8.25	32.0	
1:35:00	1.70	8.50		
1:40:00	1.70	17.00	32.1	
1:50:00	1.70	17.00	32.1	
2:00:00	1.70	8.50	32.1	
2:05:00	1.70	17.00	32.1	
2:15:00	1.70	17.00	32.1	
2:25:00	1.70	12.08	32.1	
2:32:00	1.75	8.75	32.1	
2:37:00	1.75	6.98	32.0	
2:41:00	1.74	8.75	31.9	
2:46:00	1.76	10.56	31.7	
2:52:00	1.76	7.02	31.7	
2:56:00	1.75	3.50	31.7	
2:58:00	1.75	3.50	31.9	
3:00:00	1.75		31.7	
Sum				27.4 liters (as air)
Average Flow Rate				152 ml/min
SpGr Correction Factor				1.048 dimensionless multiplier
Sample Temperature				0.01 Celcius
Total Sample Volume				28.7 liters of biogas @STP

Mass and Concentrations

Compound	MW	Front (ug)	Back (ug)	Carryover	Total (ppmv)
Silicon	28.09	2,500	20	1%	69.9
D4	296.62	226	0	0%	0.59
D5	370.77	5,460	18	0%	11.51

Silica/Siloxane Molar Ratio

1.17

Time	Sample Flow (Hundred ml/min)	Sample Volume (Hundred ml)	Operating Data	
0:00:00	0.50	0.9	Sample Start Time	6/20/08 10:00
0:01:00	1.40	2.8	Sample End Time	6/20/08 13:00
0:03:00	1.45	1.4	Biogas Temperature	91.8 F (pipeline)
0:04:00	1.45	5.9	Biogas Pressure	14.9 inches WC
0:08:00	1.50	1.4	Booster Pump Run Time	3 hours
0:09:00	1.35	4.0	Biogas Data	
0:12:00	1.35	10.8	Specific Gravity	0.91
0:20:00	1.35	5.7	Specific Volume (STP)	22.3 liters/gmole
0:24:00	1.49	3.0	<div> <h3>Sample Flowrate</h3> <p>Flowrate (hundred ml/min)</p> <p>Sampling Time (Hours:Minutes)</p> </div>	
0:26:00	1.49	11.2		
0:34:00	1.30	8.4		
0:40:00	1.49	9.0		
0:46:00	1.50	12.0		
0:54:00	1.50	1.5		
0:55:00	1.50	9.0		
1:01:00	1.49	22.4		
1:16:00	1.49	9.0		
1:22:00	1.50	6.0		
1:26:00	1.50	21.0		
1:40:00	1.50	52.5		
2:15:00	1.50	15.0		
2:25:00	1.51	16.3		
2:36:00	1.45	11.4		
2:44:00	1.40	2.9		
2:46:00	1.49	6.0		
2:50:00	1.51	6.0		
2:54:00	1.50	4.5		
2:57:00	1.51	4.5		
3:00:00	1.50			
Sum		26.5 liters (as air)		
Average Flow Rate		147 ml/min		
SpGr Correction Factor		1.048 dimensionless multiplier		
Sample Temperature		4.0 Celcius		
Total Sample Volume		27.3 liters of biogas @STP		

Mass and Concentrations

Compound	MW	Front (ug)	Back (ug)	Carryover	Total (ppmv)
Silicon	28.09	12,800	361	3%	383.2
D4	296.62				
D5	370.77				

Time	Chilled Flow (Hundred ml/min)	Chilled Volume (Hundred ml)	Sample Temp (F)	Operating Data
0:00:00	0.90	1.15	31.7	Sample Start Time 6/20/08 10:00
0:01:00	1.40	2.80		Sample End Time 6/20/08 13:00
0:03:00	1.40	1.38	31.9	Biogas Temperature 91.8 F (pipeline)
0:04:00	1.35	5.50		Biogas Pressure 14.9 inches WC
0:08:00	1.40	1.38		Booster Pump Run Time 3 hours
0:09:00	1.35	4.05		Biogas Data
0:12:00	1.35	10.96	32.3	Specific Gravity 0.91
0:20:00	1.39	5.78	32.5	Specific Volume (STP) 22.3 liters/gmole
0:24:00	1.50	2.98	32.5	<p>Sample Flowrate</p>
0:26:00	1.48	11.12	32.7	
0:34:00	1.30	8.40	32.7	
0:40:00	1.50	9.00		
0:46:00	1.50	12.20	33.0	
0:54:00	1.55	1.52	33.2	
0:55:00	1.49	8.97		
1:01:00	1.50	22.58		
1:16:00	1.51	9.03	33.6	
1:22:00	1.50	6.00	33.6	
1:26:00	1.50	21.35	33.6	<p>Sample Flowrate</p>
1:40:00	1.55	54.25	33.9	
2:15:00	1.55	15.30	34.3	
2:25:00	1.51	16.50	34.5	
2:36:00	1.49	11.76	34.5	
2:44:00	1.45	3.00	34.3	
2:46:00	1.55	6.20		
2:50:00	1.55	6.10		
2:54:00	1.50	4.51		
2:57:00	1.51	4.53	34.7	
3:00:00	1.51		34.8	
Sum			26.8 liters (as air)	
Average Flow Rate			149 ml/min	
SpGr Correction Factor			1.048 dimensionless multiplier	
Sample Temperature			0.77 Celcius	
Total Sample Volume			28.0 liters of biogas @STP	

Mass and Concentrations

Compound	MW	Front (ug)	Back (ug)	Carryover	Total (ppmv)
Silicon	28.09	3,360	193	6%	100.8
D4	296.62				
D5	370.77				

Time	Sample Flow (Hundred ml/min)	Sample Volume (Hundred ml)	Operating Data
0:00:00	1.25	1.4	Sample Start Time 8/6/08 11:00
0:01:00	1.50	1.5	Sample End Time 8/6/08 14:00
0:02:00	1.50	3.0	Biogas Temperature 94.5 F (pipeline)
0:04:00	1.49	1.5	Biogas Pressure 14.2 inches WC
0:05:00	1.49	4.5	Booster Pump Run Time 3 hours
0:08:00	1.50	4.5	Biogas Data
0:11:00	1.51	1.5	Specific Gravity 0.91
0:12:00	1.51	4.5	Specific Volume (STP) 22.3 liters/gmole
0:15:00	1.51	3.0	<h3>Sample Flowrate</h3>
0:17:00	1.52	12.2	
0:25:00	1.53	6.1	
0:29:00	1.53	7.7	
0:34:00	1.53	7.7	
0:39:00	1.54	7.7	
0:44:00	1.54	10.6	
0:51:00	1.50	6.0	
0:55:00	1.50	3.0	
0:57:00	1.51	10.5	
1:04:00	1.50	1.5	
1:05:00	1.55	6.1	
1:09:00	1.51	4.6	
1:12:00	1.55	4.6	
1:15:00	1.55	15.7	
1:25:00	1.60	24.1	
1:40:00	1.61	8.1	
1:45:00	1.61	48.4	
2:15:00	1.61	17.7	
2:26:00	1.61	0.0	
2:26:00	1.61	6.4	
2:30:00	1.61	8.0	
2:35:00	1.61	16.1	
2:45:00	1.61	6.4	
2:49:00	1.61	6.4	
2:53:00	1.61	4.8	
2:56:00	1.61	6.4	
3:00:00	1.61		
Sum		28.3 liters (as air)	
Average Flow Rate		157 ml/min	
SpGr Correction Factor		1.048 dimensionless multiplier	
Sample Temperature		4.0 Celcius	
Total Sample Volume		29.2 liters of biogas @STP	

Mass and Concentrations

Compound	MW	Front (ug)	Back (ug)	Carryover	Total (ppmv)
Silicon	28.09	15,500	230	1%	428.7
D4	296.62	3,530	87	0%	9.33
D5	370.77	25,000	291	1%	52.21

Silica/Siloxane Molar Ratio

1.44

Time	Chilled Flow (Hundred ml/min)	Chilled Volume (Hundred ml)	Sample Temp (F)	Operating Data
0:00:00	1.50	1.50	33.6	Sample Start Time 8/6/08 11:00
0:01:00	1.50	1.50	34.1	Sample End Time 8/6/08 14:00
0:02:00	1.50	3.00	34.5	Biogas Temperature 94.5 F (pipeline)
0:04:00	1.50	1.50	34.7	Biogas Pressure 14.2 inches WC
0:05:00	1.50	4.50		Booster Pump Run Time 3 hours
0:08:00	1.50	4.52		Biogas Data
0:11:00	1.51	1.51	33.4	Specific Gravity 0.91
0:12:00	1.52	4.58	33.4	Specific Volume (STP) 22.3 liters/gmole
0:15:00	1.53	3.06	33.4	<h3>Sample Flowrate</h3>
0:17:00	1.53	12.16	33.2	
0:25:00	1.51	6.08	33.6	
0:29:00	1.53	7.65	33.8	
0:34:00	1.53	7.70	33.8	
0:39:00	1.55	7.75	34.1	
0:44:00	1.55	10.85	34.5	
0:51:00	1.55	6.20	33.2	
0:55:00	1.55	3.05	32.7	
0:57:00	1.50	10.94	32.7	
1:04:00	1.63	1.61	32.9	
1:05:00	1.60	6.40	33.0	
1:09:00	1.60	4.82	33.0	
1:12:00	1.61	4.82	33.2	
1:15:00	1.60	16.06	33.2	
1:25:00	1.61	24.19	33.6	
1:40:00	1.61	8.06	34.3	
1:45:00	1.61	48.38	34.3	
2:15:00	1.61	17.74	35.2	
2:26:00	1.61	0.00	35.4	
2:26:00	1.65	6.60	35.4	
2:30:00	1.65	8.31	35.4	
2:35:00	1.68	16.87	34.3	
2:45:00	1.70	6.90	34.1	
2:49:00	1.75	6.86	34.1	
2:53:00	1.68	5.00	34.1	
2:56:00	1.65	6.54	34.3	
3:00:00	1.62		34.5	
Sum				28.7 liters (as air)
Average Flow Rate				160 ml/min
SpGr Correction Factor				1.048 dimensionless multiplier
Sample Temperature				1.06 Celcius
Total Sample Volume				30.0 liters of biogas @STP

Mass and Concentrations

Compound	MW	Front (ug)	Back (ug)	Carryover	Total (ppmv)
Silicon	28.09	4,920	0	0%	130.6
D4	296.62	2,820	55	0%	7.22
D5	370.77	6,220	68	1%	12.64

Silica/Siloxane Molar Ratio

1.42

Time	Sample Flow (Hundred ml/min)	Sample Volume (Hundred ml)	Operating Data
0:00:00	2.00	8.0	Sample Start Time 12/23/08 11:11
0:04:00	2.00	6.2	Sample End Time 12/23/08 14:11
0:07:00	2.10	12.9	Biogas Temperature 77.4 F (pipeline)
0:13:00	2.20	8.9	Biogas Pressure 15.0 inches WC
0:17:00	2.25	2.1	Booster Pump Run Time 3 hours
0:18:00	2.00	2.0	Biogas Data
0:19:00	2.00	20.0	Specific Gravity 0.91
0:29:00	2.00	20.0	Specific Volume (STP) 22.3 liters/gmole
0:39:00	2.00	16.0	<p>Sample Flowrate</p>
0:47:00	1.99	12.0	
0:53:00	2.00	4.0	
0:55:00	1.99	19.9	
1:05:00	1.99	15.2	
1:13:00	1.80	1.9	
1:14:00	2.00	22.0	
1:25:00	2.00	44.1	
1:47:00	2.01	18.0	
1:56:00	2.00	24.0	
2:08:00	2.00	14.0	
2:15:00	2.00	10.0	
2:20:00	2.01	28.1	
2:34:00	2.00	28.0	
2:38:00	2.00	36.0	
2:48:00	2.00	16.0	
2:56:00	2.00	8.0	
3:00:00	2.00		
Sum			39.7 liters (as air)
Average Flow Rate			221 ml/min
SpGr Correction Factor			1.048 dimensionless multiplier
Sample Temperature			4.0 Celcius
Total Sample Volume			41.0 liters of biogas @STP

Mass and Concentrations

Compound	MW	Front (ug)	Back (ug)	Carryover	Total (ppmv)
Silicon	28.09	2,490	0	0%	48.3
D4	296.62	233	0	0%	0.43
D5	370.77	6,100	0	0%	8.96

Silica/Siloxane Molar Ratio

1.04

Time	Sample Flow (Hundred ml/min)	Sample Volume (Hundred ml)	Operating Data	
0:00:00	1.50	0.7	Sample Start Time	1/29/09 10:00
0:00:23	2.00	0.5	Sample End Time	1/29/09 13:00
0:00:39	1.80	0.6	Biogas Temperature	78.9 F (pipeline)
0:00:58	2.10	1.9	Biogas Pressure	15.3 inches WC
0:01:50	2.25	5.6	Booster Pump Run Time	3 hours
0:04:18	2.30	1.9	Biogas Data	
0:05:08	2.30	1.2	Specific Gravity	0.91
0:05:41	2.00	0.3	Specific Volume (STP)	22.3 liters/gmole
0:05:51	1.99	5.8	<div style="text-align: center;"> <h3>Sample Flowrate</h3> </div>	
0:08:46	1.99	20.2		
0:19:00	1.95	1.1		
0:19:32	2.00	6.5		
0:22:42	2.10	1.9		
0:23:39	2.00	10.7		
0:29:00	1.99	7.0		
0:32:32	2.00	7.0		
0:36:00	2.01	4.0		
0:38:00	2.00	16.7		
0:46:20	2.00	28.7		
1:00:40	2.01	90.9		
1:49:00	1.75	1.9		
1:50:00	2.00	4.0		
1:52:00	2.00	12.9		
1:58:27	1.99	10.0		
2:03:30	1.99	19.3		
2:13:09	2.00	15.0		
2:20:40	2.00	17.3		
2:28:55	2.20	1.5		
2:29:38	2.00	13.2		
2:35:55	2.20	1.6		
2:36:40	2.00	11.2		
2:42:16	1.99	15.4		
2:50:00	2.00	13.6		
2:56:48	2.00	6.4		
3:00:00	2.00			
Sum		35.6 liters (as air)		
Average Flow Rate		198 ml/min		
SpGr Correction Factor		1.048 dimensionless multiplier		
Sample Temperature		4.0 Celcius		
Total Sample Volume		36.8 liters of biogas @STP		

Mass and Concentrations

Compound	MW	Front (ug)	Back (ug)	Carryover	Total (ppmv)
Silicon	28.09	2,380	135	6%	54.4
D4	296.62	0	0	0%	0.00
D5	370.77	3,930	0	0%	6.43

Silica/Siloxane Molar Ratio

1.69

Time	Sample Flow (Hundred ml/min)	Sample Volume (Hundred ml)	Operating Data
0:00:00	1.50	0.3	Sample Start Time 3/25/09 10:00
0:00:10	1.75	0.4	Sample End Time 3/25/09 13:00
0:00:22	2.00	1.1	Biogas Temperature 81.9 F (pipeline)
0:00:55	2.15	0.5	Biogas Pressure 14.6 inches WC
0:01:10	2.00	3.2	Booster Pump Run Time 3 hours
0:02:54	1.70	0.4	Biogas Data
0:03:08	2.00	1.7	Specific Gravity 0.91
0:04:00	2.00	2.0	Specific Volume (STP) 22.3 liters/gmole
0:05:00	2.00	2.0	<p>Sample Flowrate</p>
0:06:00	2.00	4.0	
0:08:00	2.04	6.1	
0:11:00	2.01	8.0	
0:15:00	2.01	18.2	
0:24:00	2.04	3.0	
0:25:30	2.00	11.8	
0:31:49	1.75	0.3	
0:32:00	2.00	14.0	
0:39:00	2.00	66.7	
1:12:00	2.04	38.9	
1:31:00	2.05	22.6	
1:42:00	2.05	10.2	
1:47:00	2.04	10.2	
1:52:00	2.05	45.1	
2:14:00	2.05	47.3	
2:37:00	2.06	37.0	
2:55:00	2.05	10.3	
3:00:00	2.05		
Sum		36.5 liters (as air)	
Average Flow Rate		203 ml/min	
SpGr Correction Factor		1.048 dimensionless multiplier	
Sample Temperature		4.0 Celcius	
Total Sample Volume		37.7 liters of biogas @STP	

Mass and Concentrations

Compound	MW	Front (ug)	Back (ug)	Carryover	Total (ppmv)
Silicon	28.09	1,310	0	0%	27.6
D4	296.62	0	0	0%	0.00
D5	370.77	2,280	0	0%	3.64

Silica/Siloxane Molar Ratio

1.52

Time	Sample Flow (Hundred ml/min)	Sample Volume (Hundred ml)	Operating Data
0:00:00	2.00	3.0	Sample Start Time 5/29/09 10:20
0:01:30	2.00	3.9	Sample End Time 5/29/09 13:20
0:03:30	1.90	1.0	Biogas Temperature 88.7 F (pipeline)
0:04:00	2.00	12.0	Biogas Pressure 14.9 inches WC
0:10:00	2.00	4.0	Booster Pump Run Time 3 hours
0:12:00	2.00	6.0	Biogas Data
0:15:00	2.00	4.0	Specific Gravity 0.91
0:17:00	2.00	40.0	Specific Volume (STP) 22.3 liters/gmole
0:37:00	2.00	46.6	Sample Flowrate
1:00:00	2.05	2.0	
1:01:00	2.00	18.0	
1:10:00	2.00	59.5	
1:39:00	2.10	2.0	
1:40:00	2.00	12.0	
1:46:00	2.00	16.2	
1:52:00	3.40	1.4	
1:52:30	2.00	6.0	
1:56:00	1.40	0.8	
1:56:30	2.00	7.0	
2:00:00	2.00	108.0	
2:54:00	2.00	0.3	
2:54:10	2.00	1.7	
2:55:00	2.00	7.8	
2:59:00	1.90	1.0	
2:59:30	2.00	1.0	
3:00:00	2.00		
Sum			36.5 liters (as air)
Average Flow Rate			203 ml/min
SpGr Correction Factor			1.048 dimensionless multiplier
Sample Temperature			4.0 Celcius
Total Sample Volume			37.7 liters of biogas @STP

Mass and Concentrations

Compound	MW	Front (ug)	Back (ug)	Carryover	Total (ppmv)
Silicon	28.09	1,150	0	0%	24.3
D4	296.62	0	0	0%	0.00
D5	370.77	1,520	0	0%	2.43

Silica/Siloxane Molar Ratio

2.00

Constants

Air Composition	MW	Percent
Nitrogen	28.01	78.0840%
Oxygen	32.00	20.9476%
Argon	39.95	0.9340%
Carbon Dioxide	44.01	0.0314%
Neon	20.18	0.0018%
Methane	16.04	0.0002%

Gas Constant (British units)	0.7302 ft ³ atm/lbmolR
Gas Constant (metric units)	0.08206 Latm/molK
Air Compressibility Factor	0.9999
Air Molecular Weight	28.96
Biogas Compressibility Factor	0.9969
Biogas Molecular Weight	26.29
Room temperature	64.95 F

**APPENDIX 7 – TOTAL SOLIDS AND VOLATILE SOLIDS ANALYTICAL
PROCEDURE**

Narragansett Bay Commission Laboratory SOP:

Total, Fixed & Volatile Solids in Solid & Semisolid Samples

Approved By Laboratory Manager: _____

Approved By Assistant Laboratory Manager: _____

Approved By Chemist or Technician: _____

Date Approved: _____

GENERAL

This method is applicable to the determination of total solids and its fixed and volatile fractions in such solid and semisolid samples as river and lake sediments, sludge separated from water and wastewater treatment processes, and sludge cakes from vacuum filtration, centrifugation, or other sludge dewatering processes.

SAMPLE HANDLING AND PRESERVATION

Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration to 4°C, to minimize microbiological decomposition of solids, is recommended. Analyze within 24 hours of collection. Maximum holding time is 7 days.

INTERFERENCES

These determinations are subject to negative error due to loss of ammonium carbonate and volatile organic matter during drying, especially with sediments, sludges and sludge cakes. The mass of organic matter recovered from sludge and sediment require a longer ignition time than that specified for wastewaters, effluents, or polluted waters. Carefully observe specified ignition time and temperature to control losses of volatile organic salts if these are a problem. Make all weighing quickly because wet samples tend to lose weight by evaporation. After drying or ignition, residues often are very hygroscopic and readily absorb moisture from the air.

APPARATUS

- Porcelain evaporating dishes
- Heating block, range setting of 103-105°C and drying oven set at 103 - 105°C
- Muffle furnace, set @ 500°C $\pm$ 50°C
- Balance (top loader) minimum weighing 0.01 mg.
- Desiccator, desiccant must be blue indicating no moisture.
- Pipets

QA/QC

A duplicate sample must be analyzed for TS and VS everyday. The duplicate can be of any of the samples processed on a given day. Mid range standard is ran once a week. The duplicates should agree within 5% of their average.

PREPARATION BEFORE PROCEDURE

1. Total solids- Heat dishes in drying oven for one hour.
2. Volatile solids- Ignite dishes in muffle furnace for one hour.

After removal from oven or muffle furnace cool in desiccator, weigh (W₁), and store in desiccator until ready for use.

PROCEDURE

TOTAL SOLIDS

Fluid samples: A (WP, ERA & Pressate)

Pipet a measured Volume of a well mixed samples into dishes.

Fluid samples: B

1. Total solids- Heat dishes in drying oven for one hour.
2. Place 25 to 50 gram of a well mixed samples in dish and weigh (W₂) in grams.
3. Place dish on heating block and evaporate to dryness overnight. Place in oven @ 105° for one (1) hour. Place in desiccator and weigh. Record the mass as W₃, in grams.
4. Compute the percent of total solids (Equation 1). Duplicates should agree within 5% of their average.

Solid samples: If the sample consists of discrete pieces of solid material (dewatered sludge, for example), take cores from each piece with a No. 7 cork borer or pulverize the entire sample coarsely on a clean surface by hand, using rubber gloves. Then proceed to steps 1 to 3.

FIXED & VOLATILE SOLIDS

1. Place dish with the dried residue from step 3 in cool muffle furnace and ignite for 1 hour until sample is burned completely. Length of time for complete burning depends on size of sample. If unsure that constant mass has been obtained, record mass from first heating, then reheat and weigh again in the same manner until a constant mass reading obtained. The weight change should be less than 4% or 50 mg, whichever is less.
2. Allow the dish to partially cool (~10-15 minutes) in air until most of the heat has dissipated; then cool in a desiccator to room temperature. Weigh the dish and record as W₄, in grams. The loss of weight (W₃-W₄) is the volatile matter. The percent volatile matter may then be computed (Equation 2). Duplicate determinations should agree within 5% of their average.

CALCULATION

$$\text{Percent total solids} = \frac{\text{mass of dry solids } (W_3 - W_1) \times 100}{\text{mass of wet sludge } (W_2 - W_1)} \quad (1)$$

Percent volatile matter

$$\frac{\text{mass of volatile solids } (W_3 - W_4) \times 100}{\text{mass of dry sludge } (W_3 - W_1)} \quad (2)$$

$$\text{Percent fixed matter} = \frac{\text{mass of ash } (W_4 - W_1) \times 100}{\text{mass of dry sludge } (W_3 - W_1)} \quad (3)$$

Where

- All weights are expressed in grams
- W_1 =mass of the prepared evaporating dish
- W_2 =mass of the prepared dish and wet sample
- W_3 =mass of the dish and sample after the drying step
- W_4 =mass of the dish and sample after the ignition step

REFERENCE

Standard Methods, 2540 G.; 2540B

**APPENDIX 8 – TOTAL SUSPENDED SOLIDS AND VOLATILE SUSPENDED
SOLIDS ANALYTICAL PROCEDURE**

Narragansett Bay Commission Laboratory SOP:

Total Suspended Solids (TSS) / Volatile Suspended Solids (VSS)

Approved By Laboratory Manager: _____

Approved By Assistant Laboratory Manager: _____

Approved By Chemist or Technician: _____

Date Approved: _____

GENERAL

To determine the concentration of material retained by a 1.5 micron pore size glass fiber filter (934-AH) in wastewater samples. For special studies of Rivers and Bays use 47 mm with .70 pore filters with Grade F.

SAMPLE HANDLING AND PRESERVATION

Samples should be analyzed as soon as possible. If analysis is delayed, store the sample in the main laboratory refrigerator @ 4°C. Maximum holding time is 7 days. Bring samples to room temperature before analysis.

INTERFERENCES

Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not desired in the final result. Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200-mg residue.

APPARATUS

- Membrane filter apparatus
- Vacuum pump
- Vacuum flask
- Drying oven, 103° - 105°C
- Muffle furnace, controlled at 550°C, $\pm 50^\circ\text{C}$
- Analytical balance
- Glass fiber filter disks, without organic binder, 1.5 microns
- Glass fiber filter disks, Grade F, .7 pore filters
- Desiccator
- Graduated cylinders; volumetric pipets

STANDARD PREPARATION (Standard may be commercially prepared)

Check solutions:

1. Mid range Standard
2. High Standard

SAMPLE SELECTION

All legal, DMR and standard samples must be run in triplicate. Other samples may be run in duplicate or singly as needed.

PROCEDURE

1. Place a glass fiber filter disk rough side up in the filter holder apparatus.
2. Apply vacuum and wash the disk with three successive 20mL portions of deionized water. Remove the filter disks from the filter holder apparatus and transfer to an aluminum dish.
3. Dry the disk at 104°C for 1 hour or until constant mass is attained, or until the weight loss is less than 4% of the previous weight or 0.5mg, whichever is less. If volatile solids are to be determined, ignite container and disk at 550°C for 20 minutes. Allow the crucible to partially cool in air until most of the heat has dissipated; then cool in a desiccator to room temperature. Weight the disk immediately before use. Record this mass as W_1 , in grams.
4. Shake the sample vigorously and transfer the desired volume to a graduated cylinder (or use a volumetric pipet). Filter the measured volume of a sample through the filter disk with a vacuum pump, until all traces of water have passed through. With the suction on, rinse the graduated cylinder, filter funnel, wall and filter with three successive portions of deionized water, until all the water has passed through. Carefully remove the filter from the filter apparatus and transfer to the container. Dry at 103° to 105°C for at least 1 hour (some sample matrixes may require longer dry times, such as RAS and MXL); cool in a desiccator to room temperature; weigh, repeat cycle of drying, cooling desiccating and weighing until a constant weight is obtained or until weight change is <4% or 0.5mg and record as W_2 , in grams.
5. For volatile suspended solids determination, place the previously weighed sample from Step 4, in a muffle furnace at 550°C for 5-20 minutes. Allow the dish to partially cool in air until most of the heat has been dissipated, then cool in a desiccator to room temperature and weigh and record as W_3 , in grams.

CALCULATIONS

mg/L Total Suspended Solids =

$$\frac{(W_2 - W_1) \times 1000 \text{mL/L} \times 1000 \text{mg/g}}{\text{mL of sample filtered}}$$

mg/L Volatile Suspended Solids =

$$\frac{(W_2 - W_3) \times 1000 \text{mL/L} \times 1000 \text{mg/g}}{\text{mL sample}}$$

Where

W_1 , W_2 and W_3 are expressed in grams.

W_1 = mass of the prepared filter pad

W_2 = mass of filter pad after the sample filtration step, and

W_3 = mass of filter pad after the ignition step.

QA/QC

- Samples results must agree within ±20% of each other.
- Certified Mid Standard must be run daily.
- Certified High Standard must be run daily.

REFERENCE

Standard Methods, 2540D, 2540E

APPENDIX 9 – SILOXANE SCREENING ANALYTICAL PROCEDURE

Narragansett Bay Commission Laboratory SOP:

Siloxane Analysis

Approved By Laboratory Manager: _____

Approved By Assistant Laboratory Manager: _____

Approved By Chemist or Technician: _____

Date Approved: _____

DISCUSSION

A Purge and Trap/Gas Chromatographic/Mass Spectrometric procedure is used for the analysis of octamethyltetrasiloxane (D4) and decamethylpentasiloxane (D5). Purge and trap analysis is not an approved method for the analysis of siloxanes as these compounds are almost insoluble in water. This method is only used to tentatively identify and quantify these two siloxanes at very high concentrations. A standard curve is formed using Aldrich standards D4 (98%) and D5 (97%). A mixed siloxane standard of D4 (196 mg/L) and D5 (776 mg/L) dissolved in methanol is used.

The curve ranges from 78 ug/L to 624 ug/L (D4) and from 310 ug/L to 2480 ug/L (D5).

The sample purger is designed to accept 5 ml samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap has a total volume of less than 20 ml. The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin.

The trap is at least 25 cm long and has an inside diameter of at least 0.105 inch. The trap is packed to contain the following minimum lengths of absorbents:

Tenax : 24 cm (Purge Trap A, Supelco).

The desorber is capable of rapidly heating the trap to 220°C and performing the bake out step at 260°C.

The volatiles then pass into the GC unit where the resolved analytes elute and are then passed into the MS for identification and quantification.

Methylene Chloride contaminates the EPA 624 GC/MS. Do not use Methylene Chloride to make standards or any other reagents. Purge & Trap grade Methanol is the only solvent that is to be used in preparing standards and other reagents for the EPA 624 GC/MS.

SAMPLE HOLDING TIME & PRESERVATION

The siloxane samples are collected in plastic containers and kept refrigerated following collection.

INTERFERENCES

Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system is demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks.

Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during storage and handling.

Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carryover, the purging device and sampling syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a blank to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high purgeable levels, it may be necessary to wash out the purging device with methanol, rinse it with distilled water, and then dry it in a 105°C oven between analysis. The trap and other parts of the system are also subject to contamination, therefore, frequent bakeout and purging of the entire system may be required.

APPARATUS & MATERIALS

Syringes: 10 ul, 25 ul, 50 ul, 100 ul

Class A Volumetric Flasks: 10 ml, 100 ml

Vials: 40 ml with Teflon screw cap top.

Purge & Trap Unit: Tekmar Velocity XPT Sample Concentrator

Gas Chromatograph: (Clarus 500) A complete analytical system with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, and gases.

Column: 60 M x 0.025 mm ID, 1.4 um df. Perkin-Elmer Elite 624.
Maximum programmable temp. 240 C

Mass Spectrometer: (Clarus 500) Capable of scanning from 35 to 300 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode. It is auto tuned to produce a mass spectrum, which meet all the criteria listed in Table #1 below, when 50 ng of 4-bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

Data System: Turbomass Ver 4.5. This system is interfaced to the mass spectrometer and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer's software allows the user to search any GC/MS data file for ions of a specified mass and plotting of ion abundances versus time or scans number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software is also capable of integrating the abundance in any EICP between specified time or scan number limits.

PURGE & TRAP

All samples must be allowed to warm to ambient temperature before analysis.

The following parameters must be set for normal volatile analysis:

Line Temp	150 C
Valve Temp	150 C
Mount Temp	90 C
MCS Line Temp	NA
Purge Ready Temp	33 C
Dry flow standby temp	215 C
Standby flow	10 ml/min.
Pre-purge time	0.00 min.
Pre-purge flow	40 ml/min.
Sample Heater	Off
PrePurge Time	3 minutes
Sample PreHeat Time	1 minutes
Sample PreHeat Temp	40 C
Purge Temp	0 C
Purge Time	11 minutes
Dry Purge Time	0.50 minutes
GC Start	Start of Desorb
Desorb preheat temp	210 C
Desorb Temp	215 C
Desorb Time	1 minutes
Desorb Drain	On
Desorb flow	200ml/min.
Bake Time	9.00 minutes
Bake Temp	225 C
Dryflow bake temp	215 C
Bake flow	300 ml/min.

The analyst must assure that the correct method for wastewater analysis is loaded in the VOC **Teklink 2.3** program file. To load the method file:

Click the **Teklink** icon. Click **file**, then click **open**. Choose **Method**, then choose **SII100206b.mvsa**

REAGENTS

All reagents are commercially available at concentrated levels in methanol. All dilutions are prepared with a purge and trap grade of methanol. The data concerning the preparation of standards and diluted reagents is dated and logged into a reagent data book.

PROCEDURE:

Tune:

From main turbomass window, open tune page by clicking in third button down on left side of screen (see spectacles icon).

Choose file-open. Open tune file currently in use.

Click on [press for operate] button to turn on filament.

The filament should be turned on at least 30 minutes prior to turning the reference gas on and examining masses 69,131, 219 and 502.

Choose gas-reference gas on. Masses 131 and 219 should be in the 30-35 % range compared to 69 at 100 %, and 502 should be in the 2-3% range.

Click on gas-reference gas off, and then click on [pump out reference gas].

When peaks have vanished, unclick [pump out reference gas].

To perform BFB test:

In the main turbomass window, choose file-open project. From the c:\ directory, choose BFBTUNE, and the project BFBTUNE.PRO. A sample table labeled BFB#.SPL should appear. Choose Samples-load format-BFB format. Choose Samples-Add to add one sample. The file name for the daily BFB run should be BFBmmdyyA. If the run must be repeated, the next file name would be BFBmmdyyB.

Confirm that the MS tune file listed is the most current file. To change the MS method, MS tune, and GC method files, left-click in the cell, and use the drop-down list to choose another file. Right-click in the GC method file cell and choose [open]. Choose equilibrate and OK.

To begin run, highlight the sample row, and press right arrow button in the toolbar. A green light will appear next to the number of the sample row. The general status box will display "setting up", then "ready". When the starting temperature is reached, the GC status box will display "pre-run", then "ready". For injections, use the touch screen on the GC. Touch the icon of a syringe, and wait for the "ready" display. Touch the start button after filling the syringe with 1 ul of BFB, and wait until the 5 second countdown is complete, and a beep is heard. Inject the sample. The run will start. After the solvent delay is complete, the chromatogram can be viewed by choosing View-Chromatogram while the active sample row is highlighted.

A passing Tune must be obtained to process samples. The following Criteria must be met for BFB:

<u>Mass</u>	<u>Ion Abundances Criteria</u>
50	15 to 40 % of mass 95
75	30 to 60 % of mass 95
95	base peak, 100 % of relative abundance
96	5 to 9 % of mass 174
173	less than 2 % of mass 174
174	greater than 50 % of mass 95
175	5 to 9 % of mass 174
176	greater than 95 % but less than 101% of mass 174
177	5 to 9 % of mass 176

Blank Analysis:

Click the Turbomass Icon.

Click the Teklink icon. Click **OK**.

In the main turbomass window, choose file-open project. In the turbomass directory, choose EPA624.PRO. To open a particular sample list file, use file-open, and choose the file name. Open the tune page and confirm that the heading includes the current project and correct tune file. Turn on the filament by clicking on [press for operate]. The filament should be on for at least one hour before running samples.

Either use a previous sample list and add a sample to it, or use the list as a template, copying the last row to a new sample list file. Check to confirm that the correct MS method, MS tune and GC method files are active. Open the GC method file and equilibrate. Use BlmmddyA as the blank file name. A blank must be run daily before running samples. If another blank must be run during the same day, the file name should be BlmmddyB. Use the LIMS number of a plant or industrial sample as the file name. Under Sample ID, either identify the plant sample (ex. BPINF 11/03/03) or use the EMDA number and the date to identify industrial samples. If the sample has been diluted, enter the dilution factor in the user factor column.

Highlight the sample row and press start. The GC temperature should be no higher than 49 C (in the case where the starting temperature is 35 C) when the purge and trap program is begun.

A sample blank must be analyzed before plant or industrial samples. The blanks should indicate little or no residual compounds. If there are high concentrations of residual compounds present, the analyst must thoroughly flush the purge and trap frit sparger, and clean the 5 ml syringe.

Using carbon free water, pour water into a 5 ml syringe short of overflowing. Adjust the syringe to the 5 ml mark, making sure no air is present in the syringe. Using a 25 ul syringe, draw 20 ul of 5 ppm Internal Standard/System Monitoring Compounds Mix (IS/SMC). Inject the 20 ul of IS/SMC mix into the 5 ml syringe containing carbon free water. Inject the 5 ml sample into the Purge & Trap concentrator.

Press the arrow button in the teklink screen to start purging.

Sample Analysis:

Pour sample into a 5 ml syringe short of overflowing. Adjust the syringe to the 5 ml mark, making sure no air is present in the syringe. Using a 25 ul syringe, draw 20 ul of 5 ppm Internal Standard/System Monitoring Compounds Mix (IS/SMC). Inject the 20 ul of IS/SMC mix into the 5 ml syringe containing the sample. Inject the 5 ml sample into the Purge & Trap concentrator.

Calibration:

A four-point initial calibration curve is prepared and analyzed using the instrument conditions given for sample analysis. A linear regression, using 1/x fit weighting, is performed. The response type calculated is external (absolute).

QC/QA:

Prior to running the method blank, the calibration curve must be verified each day with a QC sample. The check standard used in this method is a midrange standard from the mixed standard used for the calibration curve.

The difficulty found in establishing an adequate recovery from this standard makes it impossible to regard this analysis as providing more than qualitative results.

Data Acquisition:

To access data after the completion of an analysis:

When the run is complete, highlight the sample row or rows you wish to quantitate and click on quantify-process samples. A screen will appear in which the boxes for "integrate samples" and "quantify samples" should be checked. The method listed is the qualitative method used to identify the compounds according to mass and retention time. The curve listed is the curve formed from the most recently run set of calibrants for this method. If these entries are correct, click OK.

To view the results, click on Quantify-view results. The summary will appear in the same order as that of the compound list in the method.

Although only one sample row may have been chosen for quantification, all the rows in the sample list will appear. Left-click twice on a sample row to see the peak of that compound, with retention time, mass, and abundance information. The peak can then be integrated to check on the accuracy of the identification.

REPORTS

From the Quantify-view results screen, results can be exported to an excel spreadsheet using the "copy summary by compound" or the "copy summary by sample" command. The former would be used for calibration curve files, and the latter for sample files. If a sample has been diluted, and the dilution factor entered in the user factor column, the concentrations of the internal standards and the system monitoring compounds will also be multiplied by that factor.

The entire sample list data file will be exported to the excel workbook.

Copy the current files to the second sheet of the workbook. Save the workbook under the name Gcresultsmmddy.xls. Choose file-new and select the Report template II. Save the template as an excel spreadsheet file using the LIMS ID # as the file name. Copy the sample ID row from the Gcresults spreadsheet to the report template. Copy the retention times of the internal standards, and the concentrations and retention times of the system monitoring compounds, to the template. Copy the concentrations and retention times of all compounds present in the target list to the template. If a dilution was performed, enter the dilution factor in the sample information section. Divide the concentrations of the system monitoring compounds by that factor. Add a column to the right of the detection limit column in which the detection limit is multiplied by the dilution factor.

Compare concentration values to the detection limits listed in the template, and fill in the concentration list with ND where the concentration is below the detection limit. Save the file. Save the file to the network drive (in the EPA 624 folder). Print two copies of the file. Keep one copy in the appropriate folder, and deliver the other to the lab manager. Save a copy of the raw data file to the Clarus data folder on the network drive.

DATA INTERPRETATION

Qualitative Analysis:

An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum) obtained on the

user's GC/MS. These standard reference spectra may be obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC relative retention time (RRT) as those of the standard component; and (2) correspondence of the sample component and the standard component mass spectra.

The quantification is made on the basis of the calibration data previously assembled, in which the area of the target ion of a given analyte is relative to the area of the internal standard target ion. Unknown peaks can tentatively be identified by a computer search of the 75 K library of the NIST, which is accessed through the F1 function key.

Data Control:

After a sample run is complete and the sample raw data is generated, the analyst reviews the raw data.

When an analyte exceeds the permitted limit for Plant and Industrial samples, the Laboratory Manager must be notified and a duplicate sample must be run to confirm results.

Atlantic Analytical and NBC Lab Results From May/June 2007 Samplings											
AAL Siloxane Results (ppb)					NBC (ppm and siloxane in ppb)						
Sample Location	Sample #	Aqueous (ug/l)	NBC Sample #	Total (ug/l)	K _p (L/g)	Analysis Date	Sample #	Aqueous	TSS	VSS	Analysis Date
Digester	0705-BPSPST-0037	79	0706-BPSPST-0001	75,000	29	6/18/07	0705-BPSPST-0035&836	957	39,170	33,125	5/31/07
BVI Influent	0706-BPSPST-0008	97	0706-BPSPST-0006	2,770	251	6/18/07	0706-BPSPST-0010	1,514	125	110	6/26/07
KIK	0706-BPSPST-0009	57,000	0706-BPSPST-0007	340,000	13	6/18/07	0706-BPSPST-0011	17,384,772	580	377	6/26/07
FP Influent	0706-FPSPST-0006	nd	0706-FPSPST-0005	19	na	6/18/07	0706-FPSPST-0007	nd	124	107	6/26/07

Atlantic Analytical and NBC Lab Results From May/June 2007 Samplings											
AAL Siloxane Results (ppb)						NBC (ppm and siloxane in ppb)					
Sample Location	Sample #	Aqueous (ug/l)	NBC Sample #	Total (ug/l)	K _p (L/g)	Analysis Date	Sample #	Aqueous	TSS	VSS	Analysis Date
Digester	0705-BPSPST-0037	79	0706-BPSPST-0001	75,000	29	6/18/07	0705-BPSPST-0035&836	957	39,170	33,125	5/31/07
BVI Influent	0706-BPSPST-0008	97	0706-BPSPST-0006	2,770	251	6/18/07	0706-BPSPST-0010	1,514	125	110	6/26/07
KIK	0706-BPSPST-0009	57,000	0706-BPSPST-0007	340,000	13	6/18/07	0706-BPSPST-0011	17,384,772	580	377	6/26/07
FP Influent	0706-FPSPST-0006	nd	0706-FPSPST-0005	19	na	6/18/07	0706-FPSPST-0007	nd	124	107	6/26/07

**APPENDIX 10 – COMMERCIAL LABORATORY CERTIFICATES OF
ANALYSIS ARRANGED BY DATE**

Analysis	Laboratory	Analytical Method	Sample Date	D4	D5	Silicon	Other	Units
Siloxanes in Biogas	Rhode Island Analytical Laboratory	Method 8270C	12/13/2000	12	29	-	-	ppmv
Silica Particle Size	Liberty Mutual Insurance Group	Modified NIOSH 7400	6/7/2005	-	-	-	5 micron	ppmv
Siloxanes in Biogas	Air Toxics LTD	Method 8270C	8/2/2005	4.9	32.7	-	-	ppmv
Siloxanes in Sludge	Atlantic Analytical Laboratory	Extractive Method (Parker)	6/7/2007	-	-	-	various	mg/l
Siloxane in Condensate	Narragansett Bay Commission	Modified EPA Method 624	11/20/2007	-	-	-	6,478	mg/l
Siloxanes in Biogas	Air Toxics LTD	Method 8270C	12/5/2007	0.77	16	-	-	ppmv
Siloxanes in Biogas	Air Toxics LTD	Method 8270C	12/20/2007	0.52	9.5	-	-	ppmv
Siloxanes and Total Silicon in Biogas	ESS Laboratory	Method 8270, 3111D	1/25/2008	0.6	11.8	119.7	-	ppmv
Silica Thermal Conductivity	EMSL Analytical, Inc.	ASTM Method dated 11/1/07	6/16/2008	-	-	-	0.049	W/mC
Silica in Biogas	ESS Laboratory	Method 8270, 3111D	6/20/2008	-	-	383.2	-	ppmv
Siloxanes and Total Silicon in Biogas	ESS Laboratory	Method 8270, 3111D	8/6/2008	9.3	52.2	428.7	-	ppmv
Siloxanes and Total Silicon in Biogas	ESS Laboratory	Method 8270, 3111D	12/23/2008	0.4	9	48.3	-	ppmv
Siloxanes and Total Silicon in Biogas	ESS Laboratory	Method 8270, 3111D	1/29/2009	0	6.4	54.4	-	ppmv
Siloxanes and Total Silicon in Biogas	ESS Laboratory	Method 8270, 3111D	3/25/2009	0	3.6	27.6	-	ppmv
Siloxanes and Total Silicon in Biogas	ESS Laboratory	Method 8270, 3111D	5/29/2009	0	2.4	24.3	-	ppmv
Biogas Net Heating Value	Rhode Island Analytical Laboratory	ASTM D5373	6/3/2009	-	-	-	540.3	Btu/cf
Fats, Oil and Grease in Wastewater	Narragansett Bay Commission	Method 5520B	various	-	-	-	various	mg/l
Siloxanes in Wastewater	Narragansett Bay Commission	Modified EPA Method 624	various	-	-	-	various	mg/l

Liberty Mutual Certificate of Analysis

Sample Date 6/7/2005

Analysis Results

Date: Monday, June 13, 2005

James W. Skrabak, CIH
Camp Dresser & McKee Inc.
One Cambridge Place, 50 Hampshire St.

Cambridge, MA 02139,

Collector: James W. Skrabak

Sample Set: 0506072

Liberty Mutual Insurance Group
Commercial Professional Services
Industrial Hygiene Laboratory / 01C1

71 Frankland Road
Hopkinton, Ma. 01748
(800) 230-6263 FAX: (508) 435-3575

Survey Date: 6/7/2005
Date Received: 6/8/2005
Customer PO:
Project ID:
Project Number:
Project Name:
Project Location:

LM			Sampling Time (min.)	SamplingRate (liters/min)	Volume (liters)	Results		
Sample No.	Sample No.	Analyte						
0506072-001	Bulk 1	% Quartz Particle Size					<1.2 % see comments %	
	Analyte % Quartz Particle Size	Media Bulk Bulk	LOQ 10 ug/sample N/A	Method NIOSH 7500 Modified NIOSH 7400	*Modify No Yes	Instrument XRD PCM	Blank Corrected No No	Analysis Date 13-jun-05 10-jun-05

* Modify: A "Yes" indicates a minor modification in the

Lab Comments
Eva Longo
For the bulk sample: >86% of the number of particles are <5-um in diameter, 7% of the particles the particles are in the range of 10 to 20 um in diameter and <2% of the number of particles are >20 um in diameter. Note: this is NOT percent by weight.

are in the range of 5 to 10 um in diameter, 5% of

Analyzed by:

Peer Reviewed by: Kenneth Muzal

Analyzed by: Mien Peng

Peer Reviewed by: Kenneth Muzal

Approved By: Ethel Patricio

AIHA Accredited Laboratory ID

Page 1 of 2

Sample Set: 0506072

<i>Sample No.</i>	<i>Sample No.</i>	<i>Analyte</i>	<i>Sampling Time (min.)</i>	<i>SamplingRate (liters/min)</i>	<i>Volume (liters)</i>	<i>Results</i>
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Method.

Air Toxics LTD Certificate of Analysis

Sample Date 8/2/2005

Applied Filter Technology

Page 2 of 2

19524 75th Avenue S.E.
Snohomish, WA 98296
Fax (360) 668-7017

Phone (360) 668-6021

Cell (425) 232-6021

SAG™ Biogas Siloxane Removal

SAG™ TVOCs™ Analytical Summary

Project: Bucklin Point WWTF - RI

Report Date: 08/17/2005

VOCS				Test Date: 8/4/2005			
(Modified EPA Method TO-14A + Top 10 TICs - Vinyl Acetate excluded)				TICs (Tentatively Identified Compounds)			
Sample ID: 025421-0335	ppbv	Rpt. Limit	ppbv	Rpt. Limit	CAS No.	ppbv	
Freon 12	not detected	25	1,2,4-Trimethylbenzene	not detected	555-75-3	5100	N J
Freon 114	6900	25	1,3-Dichlorobenzene	not detected	580-21-4	9500	N J
Vinyl Chloride	not detected	25	1,4-Dichlorobenzene	29	1071-26-7	4900	N J
Bromomethane	not detected	25	alpha-Chlorotoluene	not detected	556-67-2	9000	N J
Chloroethane	not detected	25	1,2-Dichlorobenzene	not detected	3522-94-9	2400	N J
Freon 11	not detected	25	1,3-Butadiene	not detected	629-50-5	3600	N J
1,1-Dichloroethene	not detected	25	Hexane	not detected	0-00-0	46000	N J
Freon 113	not detected	25	Cyclohexane	not detected	594-38-7	2900	N J
Methylene Chloride	not detected	25	Bromodichloromethane	not detected	630-03-5	2700	N J
1,1-Dichloroethane	not detected	25	Dibromochloromethane	not detected	17302-28-2	2500	N J
dis-1,2-Dichloroethane	300	25	Heptane	not detected			
Chloroform	not detected	25	Cumene	not detected			
1,1,1-Trichloroethane	not detected	25	Propylbenzene	not detected			
Carbon Tetrachloride	not detected	25	Chloromethane	not detected			
Benzene	not detected	25	1,2,4-Trichlorobenzene	not detected			
1,2-Dichloroethane	not detected	25	Hexachlorobutadiene	not detected			
Trichloroethene	not detected	25	Acetone	54			
1,2-Dichloropropane	not detected	25	Carbon Disulfide	1600			
dis-1,3-Dichloropropene	not detected	25	2-Propanol	60			
Toluene	740	25	trans-1,2-Dichloroethene	not detected			
trans-1,3-Dichloropropene	not detected	25	2-Butanone (MEK)	not detected			
1,1,2-Trichloroethane	not detected	25	Tetrahydrofuran	not detected			
Tetrachloroethene	36	25	1,4-Dioxane	not detected			
1,2-Dibromoethane (EDB)	not detected	25	4-Methyl-2-pentanone	40			
Chlorobenzene	not detected	25	2-Hexanone	not detected			
Ethyl Benzene	not detected	25	Bromoform	not detected			
m,p-Xylene	67	25	4-Ethyltoluene	not detected			
o-Xylene	not detected	25	Ethanol	not detected			
Styrene	not detected	25	Methyl tert-butyl ether	not detected			
1,1,2,2-Tetrachloroethane	not detected	25	3-Chloropropene U J	not detected			
1,3,5-Trimethylbenzene	not detected	25	2,2,4-Trimethylpentane	30			

E = Exceeds Instrument Calibration Range
J = Estimated Value
N = Identification is based on assumptive data
ND = Not Detected

Applied Filter Technology

Page 1 of 2

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 Fax (360) 668-7017

Phone (360) 668-6021

SAG™ Biogas Siloxane Removal

SAG™ TVOCs™ Analytical Summary

Project: Bucklin Point WWTF - RI

Report Date: 08/17/2005

Siloxanes (By SIL-1 Sampling Method and GC/MS Analysis)				Sample Date: 8/2/2005		Test Date: 8/8/2005	
ND = Not Detected				Sample ID:		Effluent A	
Reporting Limit ppbv				µg/m ³		µg/m ³	
Pentamethyldisiloxane	47 (Interpreted by AFT)	not detected	ppbv	not detected		—	
Hexamethyldisiloxane (MM)	43 (Interpreted by AFT)	not detected	ppbv	not detected		—	
Hexamethylcyclotrisiloxane (D3)	32 (Interpreted by AFT)	not detected	ppbv	not detected		—	
Octamethyltrisiloxane (MDM)	29 (Interpreted by AFT)	not detected	ppbv	not detected		—	
Octamethylcyclotetrasiloxane (D4)	23 (Interpreted by AFT)	4900	ppbv	59500		—	
Decamethylcyclopentasiloxane (D5)	18 (Interpreted by AFT)	32700	ppbv	496000		—	
Dodecamethylcyclohexasiloxane (D6)	16 (Interpreted by AFT)	not detected	ppbv	not detected		—	
Effluent B				µg/m ³		µg/m ³	
ppbv				ppbv		ppbv	
µg/m ³				µg/m ³		µg/m ³	
Effluent B				ppbv		ppbv	
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Effluent B				ppbv		ppbv	
µg/m ³							

SILOXANE SAMPLING PROCEDURE

(Air/Gas sampling)

Summary of Procedure

The gas sample is drawn through a series of glass midget impingers containing chilled methanol. The siloxanes are trapped in the methanol. The methanol is transferred to high density polyethylene (HDPE) bottles for shipment to the laboratory.

Laboratory Provided Sampling Kit

The sampling kit consists of a specially designed cooler containing the impingers. The inlet of the first midget impinger is designed so that it can connect to a sample port using a length of Teflon tubing. The outlet of the first impinger is connected to the inlet of the second impinger and the outlet of the second impinger is in turn connected to a manual needle valve or rotameter to measure flow through the impinger. The laboratory provides pre-rinsed HDPE bottles containing 20ml of Methanol for each impinger. The contents of each bottle are poured into an impinger. The Midget impingers, each containing 20 ml of methanol, are placed in the cooler. Prior to sampling, an ice bath is set up in the cooler to chill the methanol during sampling.

Sampling Procedure

The Teflon tubing from the first impinger is connected to the source air stream. The user must adjust the flow rate on the needle valve to obtain the desired sampling rate and volume. The sampling rate and time must be noted on the Chain of Custody. After sampling, the impinger liquid is poured back into the HPDE bottles. The liquid from the impinger connected to the source should be marked as front (F) and the liquid from the back impinger should be marked as back (B). The HDPE bottles are shipped in a packed ice cooler to the laboratory under chain of custody practices.

ESS Laboratory
401-461-7181

@ AIR TOXICS LTD.

Guide for Siloxanes Sampling

Air Toxics Ltd. Method @71

Overview

This guide is intended as a resource for individuals engaged in siloxanes sampling using Air Toxics Ltd. Method @71. This guide is not a substitute for experience and cannot possibly address the multitude of actual field conditions. Method @71 is based on drawing air-phase samples through a series of midget impingers containing methanol and analyzing for dissolved siloxanes using GC/MS instrumentation. These instructions assume a pressurized sample port. A low volume air pump may be needed in some applications. This guide includes a diagram of the impinger sampling train, a parts list, step-by-step instructions, and sampling considerations.

⇒ Call Client Services at 800-985-5955 if you have any questions.

Parts List

<u>Item</u>	<u>Quantity</u>
ATL prepared methanol vials with Teflon screw caps	2
Midget impingers	2
Inert, flexible tubing	as needed
Needle valve	1
Rotameter (50-500 mL/min suggested)	1
Swagelock compression fittings	as needed
Small cooler (for ice bath and sample shipment)	1
Ice cubes (for ice bath)	as needed
Sample labels	2
Chain of Custody	1
Blue ice (for sample shipment)	as needed

Step-by-Step Sampling Instructions

- ❑ Check parts list before traveling to sample location
- ❑ Purge sample port as necessary
- ❑ Attach tubing from sample port to inlet of front impinger (see schematic)
- ❑ Attach tubing from outlet of front impinger to inlet of back impinger
- ❑ Attach tubing from outlet of back impinger to rotometer with built-in needle valve
- ❑ Set up ice bath in cooler
- ❑ Remove caps from vials and install impingers (do not over tighten)
- ❑ Place impinger/vial assemblies in ice bath
- ❑ Ensure that needle valve is initially closed
- ❑ Slowly open sample port and needle valve
- ❑ Increase flow until target flow rate is achieved (112 mL/min suggested)
- ❑ Monitor flow rate throughout sampling interval (180 minutes suggested)
- ❑ Slowly close sample port and needle valve at end of sampling interval
- ❑ Remove impingers from vials, replace caps, and tape caps in place
- ❑ Label vials FRONT and BACK (see diagram) and fill out chain-of-custody (indicate total volume sampled in liters)
- ❑ Keep vials out of sunlight and at $4 \pm 2^{\circ}\text{C}$ until ready for shipment
- ❑ Replace ice bath in cooler with blue ice
- ❑ Wrap labeled vials with aluminum foil and place in labeled Ziploc bags
- ❑ Place vials in cooler with appropriate padding
- ❑ Place chain-of-custody in a Ziploc bag and send with cooler
- ❑ Retain pink copy of chain-of-custody and relinquish samples properly
- ❑ Tape cooler shut and affix custody seal (if applicable) across lid
- ❑ Ship accordingly to meet method holding times. Method @71 has a hold time from sampling to analysis of up to 21 days. Samples must remain at $4 \pm 2^{\circ}\text{C}$.

Sampling Considerations

- Determine impinger solution volume: The user must request the laboratory to provide the desired amount of methanol in each impinger. Less impinger solution equates to lower reporting limits, but has less capacity to dissolve the target constituents. 6 mL in each vial suggested.
- Determine the sampling rate and volume: The user must determine optimum sampling rate and volume to achieve the data quality objectives of the sampling program. The amount of sample air drawn through the impingers and the amount of methanol in the impinger determine the final reporting limit concentration. The more sample air drawn through the impingers equates to more target constituent concentrated in the solution and thus lower reporting limits. Do not over sample and saturate the solution. A typical arrangement includes filling each impinger with 6 mL of methanol and sampling at a flow rate of 112 mL/min for 180 minutes. This arrangement results in a sampling volume of approximately 20

L. The resulting concentration of siloxanes that could be detected in each impinger separately would be 0.3 $\mu\text{G/L}$ (or from 16 to 49 ppbv).

- Do not sample too fast: Sampling rates from 100 to 1,000 mL/min are appropriate as long as there is not significant loss of impinger solution. Flow should bubble gas through impingers. 112 mL/min suggested.
- Maintain the rotometer in a vertical position: Ensure that the rotameter is calibrated and functioning properly. An electronic flow meter can also be used.
- Ensure flow rate is constant
- Provide total volume sampled in liters on chain-of-custody

Typical Impinger Sampling Train

Atlantic Analytical Laboratory Certificate of Analysis

Sample Date 6/7/2007

Analysis Report

Narragansett Bay Commission
One Service Road
Providence, RI 02905
401-461-8848 xt 439; (Fax: 401-784-3526)
Attn.: Linda Michel; E-mail: LMichel@narrabay.com

AAL Number: 3701
Received On: 06/11/07
Report Date: 06/20/07
PO Number: 339230

Sample ID: 8 Sewer and Treatment Plant samples (See Sample Key). For the determination of D4 and D5 Siloxanes by Hexane extraction and GC-MS analysis. Method provided by N.B.C.

Sample Key:

<u>Location</u>	<u>VOA Vials-Aqueous Siloxane</u>	<u>Amber Bottles-Total Siloxane</u>	<u>Date</u>
Digester	0705-BPSPST-0037	0706-BPSPST-0001	05/29/07
BVI Influent	0706-BPSPST-0008	0706-BPSPST-0006	06/07/07
KIK Company	0706-BPSPST-0009	0706-BPSPST-0007	06/06/07
FP Influent	0706-FPSPST-0006	0706-FPSPST-0005	06/07/07

Sample Prep:

VOA Vial Samples: 4 ml of sample was removed and replaced with 4 ml of extraction solvent. The vials were placed in an auto shaker for four hours and allowed to stand overnight. 1 ul of the solvent extract was analyzed by GC-MS.

Amber Bottles--FP and BVI Influent: 10 ml homogeneous sample aliquots were removed from respective bottles and added to 20 ml vials. 5 ml of DI water and 5 ml of extraction solvent was added. Extraction proceeded as above.

Amber Bottle--KIK Company: 4 ml aliquot of homogeneous sample was removed and added to 20 ml vial. 11 ml of DI H2O and 5 ml of extraction solvent was added and extracted as above.

Amber Bottle--Digester Sludge: 0.5 gram sample was weighed into a 20 ml vial. 15.5 ml of DI water and 5 ml of extraction solvent was added. Extraction proceeded as above.

Extraction Solvent: Hexane containing 5 ppm of Benzyl Chloride was used as the extraction solvent. The Benzyl Chloride served as an internal standard as well as a potential surrogate in event significant partitioning was observed. In all extractions, greater than 90% of the Benzyl Chloride was recovered.

GC-MS Analysis Results:

Sample # 1--Digester "Aqueous Siloxane"; 0705-BPSPST-0037

	<u>Result</u> <u>ppb w/w</u>	<u>D.L.</u> <u>ppb w/w</u>
Hexamethylcyclotrisiloxane--D3:	nd	10
Octamethylcyclotetrasiloxane--D4:	nd	10
Decamethylcyclopentasiloxane--D5:	79	10
Other Siloxanes*:	nd	10

Sample # 2--Digester "Total Siloxane"; 0706-BPSPST-0001

	Result ppb w/w	D.L. ppb w/w
Hexamethylcyclotrisiloxane—D3: -----	nd	10
Octamethylcyclotetrasiloxane—D4: -----	nd	10
Decamethylcyclopentasiloxane—D5: -----	75,000	10
Other Siloxanes*: -----	nd	10

Sample # 3—BVI Influent "Aqueous Siloxane"; 0706-BPSPST-0008

	Result ppb w/w	D.L. ppb w/w
Hexamethylcyclotrisiloxane—D3: -----	nd	10
Octamethylcyclotetrasiloxane—D4: -----	nd	10
Decamethylcyclopentasiloxane—D5: -----	97	10
Other Siloxanes*: -----	nd	10

Sample # 4—BVI Influent "Total Siloxane"; 0706-BPSPST-0006

	Result ppb w/w	D.L. ppb w/w
Hexamethylcyclotrisiloxane—D3: -----	nd	10
Octamethylcyclotetrasiloxane—D4: -----	nd	10
Decamethylcyclopentasiloxane—D5: -----	2,770	10
Other Siloxanes*: -----	nd	10

Sample # 5—KIK Company "Aqueous Siloxane"; 0706-BPSPST-0009

	Result ppb w/w	D.L. ppb w/w
Hexamethylcyclotrisiloxane—D3: -----	14	10
Octamethylcyclotetrasiloxane—D4: -----	141	10
Decamethylcyclopentasiloxane—D5: -----	57,000	10
Other Siloxanes*: -----	nd	10

Sample # 6—KIK Company "Total Siloxane"; 0706-BPSPST-0007

	Result ppb w/w	D.L. ppb w/w
Hexamethylcyclotrisiloxane—D3: -----	40	10
Octamethylcyclotetrasiloxane—D4: -----	595	10
Decamethylcyclopentasiloxane—D5: -----	340,000	10
Other Siloxanes*: -----	nd	10

Sample # 7—FP Influent "Aqueous Siloxane"; 0706-FPSPST-0006

	Result ppb w/w	D.L. ppb w/w
Hexamethylcyclotrisiloxane—D3: -----	nd	10
Octamethylcyclotetrasiloxane—D4: -----	nd	10
Decamethylcyclopentasiloxane—D5: -----	trace	10
Other Siloxanes*: -----	nd	10

Sample # 8—FP Influent "Total Siloxane"; 0706-FPSPST-0005

	Result ppb w/w	D.L. ppb w/w
Hexamethylcyclotrisiloxane—D3: -----	nd	10
Octamethylcyclotetrasiloxane—D4: -----	nd	10
Decamethylcyclopentasiloxane—D5: -----	19	10
Other Siloxanes*: -----	nd	10

Comments: * Other Siloxanes include Octamethyltrisiloxane, Decamethyltetrasiloxane, and Dodecamethylpentasiloxane.

Data from a recovery study using D4 as a "spike" was not available at the time this report was issued. Recovery data will be available at a later date.

D.L. = report detection limit. **nd** = indicates the concentration is less than the report detection limit.
-- = test not performed. **%** = percent. **ppm** = parts per million. **ppb** = parts per billion. **v/v** = volume analyte/volume sample. **w/w** = weight analyte/weight sample. **Unit Conversions:** 1 ppm v/v = 0.0001% v/v.

Reviewed By,

Richard F. Frisch

Attachments:

-none

Addendum:

-Instrument & Notebook data on-file

cc: Barry Wenskowicz; E-mail: Bwenskowicz@narrabay.com

Air Toxics LTD Certificate of Analysis

Sample Date 12/5/2007

AN ENVIRONMENTAL ANALYTICAL LABORATORY

WORK ORDER #: 0712096

Work Order Summary

CLIENT:	Mr. Russ Anderson SCS Field Services 166 Valley Street Building 3L, Suite 101 Providence, RI 02909	BILL TO:	Accounts Payable SCS Engineers 140 Route 303 Valley Cottage, NY 10989
PHONE:	401-274-4300x102	P.O. #	13207004.00T3
FAX:	401-274-4330	PROJECT #	Task 3 NBC
DATE RECEIVED:	12/06/2007	CONTACT:	Bryanna Langley
DATE COMPLETED:	12/17/2007		

<u>FRACTION #</u>	<u>NAME</u>	<u>TEST</u>
01AB	NBC-Silox. #1 Front/Back	Siloxanes
01ABB	NBC-Silox. #1 Front/Back Lab Duplicate	Siloxanes
02A	Lab Blank	Siloxanes
03A	LCS	Siloxanes

CERTIFIED BY:

Laboratory Director

DATE: 12/17/07

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Page 1 of 7

AN ENVIRONMENTAL ANALYTICAL LABORATORY

Summary of Detected Compounds SILOXANES - GC/MS

Client Sample ID: NBC-Silox. #1 Front/Back

Lab ID#: 0712096-01AB

Compound	Rpt. Limit (ppmv)	Amount (ppmv)	Rpt. Limit (uG/m3)	Amount (uG/m3)
Octamethylcyclotetrasiloxane (D4)	0.19	0.78	2300	9500
Decamethylcyclopentasiloxane (D5)	0.15	16	2300	240000

Client Sample ID: NBC-Silox. #1 Front/Back Lab Duplicate

Lab ID#: 0712096-01ABB

Compound	Rpt. Limit (ppmv)	Amount (ppmv)	Rpt. Limit (uG/m3)	Amount (uG/m3)
Octamethylcyclotetrasiloxane (D4)	0.19	0.76	2300	9200
Decamethylcyclopentasiloxane (D5)	0.15	16	2300	250000

AN ENVIRONMENTAL ANALYTICAL LABORATORY

Client Sample ID: NBC-Silox. #1 Front/Back

Lab ID#: 0712096-01AB

SILOXANES - GC/MS

File Name:	k120718	Date of Collection:	12/5/07
Dil. Factor:	2.00	Date of Analysis:	12/7/07 02:46 PM

Compound	Rpt. Limit (ppmv)	Amount (ppmv)	Rpt. Limit (uG/m3)	Amount (uG/m3)
Octamethylcyclotetrasiloxane (D4)	0.19	0.78	2300	9500
Decamethylcyclopentasiloxane (D5)	0.15	16	2300	240000
Dodecamethylcyclohexasiloxane (D6)	0.26	Not Detected	4600	Not Detected
Hexamethyldisiloxane	0.35	Not Detected	2300	Not Detected
Octamethyltrisiloxane	0.24	Not Detected	2300	Not Detected

Air Sample Volume(L): 23.4

Impinger Total Volume(mL): 27.1

Container Type: Vial

Surrogates	%Recovery	Method Limits
Hexamethyl disiloxane -d18	102	70-130

AN ENVIRONMENTAL ANALYTICAL LABORATORY

Client Sample ID: NBC-Silox. #1 Front/Back Lab Duplicate

Lab ID#: 0712096-01ABB

SILOXANES - GC/MS

File Name:	k120719	Date of Collection:	12/5/07
Dil. Factor:	2.00	Date of Analysis:	12/7/07 03:11 PM

Compound	Rpt. Limit (ppmv)	Amount (ppmv)	Rpt. Limit (uG/m3)	Amount (uG/m3)
Octamethylcyclotetrasiloxane (D4)	0.19	0.76	2300	9200
Decamethylcyclopentasiloxane (D5)	0.15	16	2300	250000
Dodecamethylcyclohexasiloxane (D6)	0.26	Not Detected	4600	Not Detected
Hexamethyldisiloxane	0.35	Not Detected	2300	Not Detected
Octamethyltrisiloxane	0.24	Not Detected	2300	Not Detected

Air Sample Volume(L): 23.4

Impinger Total Volume(mL): 27.1

Container Type: Vial

Surrogates	%Recovery	Method Limits
Hexamethyl disiloxane -d18	101	70-130

AN ENVIRONMENTAL ANALYTICAL LABORATORY

Client Sample ID: Lab Blank

Lab ID#: 0712096-02A

SILOXANES - GC/MS

File Name:	k120706	Date of Collection: NA
Dil. Factor:	1.00	Date of Analysis: 12/7/07 09:55 AM

Compound	Rpt. Limit (ppmv)	Amount (ppmv)	Rpt. Limit (uG/m3)	Amount (uG/m3)
Octamethylcyclotetrasiloxane (D4)	0.0035	Not Detected	43	Not Detected
Decamethylcyclopentasiloxane (D5)	0.0028	Not Detected	43	Not Detected
Dodecamethylcyclohexasiloxane (D6)	0.0047	Not Detected	85	Not Detected
Hexamethyldisiloxane	0.0064	Not Detected	43	Not Detected
Octamethyltrisiloxane	0.0044	Not Detected	43	Not Detected

Air Sample Volume(L): 23.4

Impinger Total Volume(mL): 1.00

Container Type: NA - Not Applicable

Surrogates	%Recovery	Method Limits
Hexamethyl disiloxane -d18	102	70-130

AN ENVIRONMENTAL ANALYTICAL LABORATORY

Client Sample ID: LCS

Lab ID#: 0712096-03A

SILOXANES - GC/MS

File Name:	k120705	Date of Collection: NA
Dil. Factor:	1.00	Date of Analysis: 12/7/07 09:31 AM

Compound	%Recovery
Octamethylcyclotetrasiloxane (D4)	100
Decamethylcyclopentasiloxane (D5)	99
Dodecamethylcyclohexasiloxane (D6)	Not Spiked
Hexamethyldisiloxane	98
Octamethyltrisiloxane	105

Air Sample Volume(L): 23.4

Impinger Total Volume(mL): 1.00

Container Type: NA - Not Applicable

Surrogates	%Recovery	Method Limits
Hexamethyl disiloxane -d18	100	70-130

Air Toxics LTD Certificate of Analysis

Sample Date 12/20/2007

AN ENVIRONMENTAL ANALYTICAL LABORATORY

WORK ORDER #: 0712493

Work Order Summary

CLIENT:	Mr. Russ Anderson SCS Field Services 166 Valley Street Building 3L, Suite 101 Providence, RI 02909	BILL TO:	Accounts Payable SCS Engineers 140 Route 303 Valley Cottage, NY 10989
PHONE:	401-274-4300x102	P.O. #	13207004.00T3
FAX:	401-274-4330	PROJECT #	TASK 3 NBC
DATE RECEIVED:	12/22/2007	CONTACT:	Bryanna Langley
DATE COMPLETED:	01/07/2008		

<u>FRACTION #</u>	<u>NAME</u>	<u>TEST</u>
01AB	NBC-Silox #2 1/2	Siloxanes
02A	Lab Blank	Siloxanes
03A	LCS	Siloxanes

CERTIFIED BY:

Laboratory Director

DATE: 01/07/08

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AN ENVIRONMENTAL ANALYTICAL LABORATORY

LABORATORY NARRATIVE

Siloxanes

SCS Field Services

Workorder# 0712493

Two Vial samples were received on December 22, 2007. The laboratory performed analysis for siloxanes by GC/MS. A sample volume of 1.0 uL was injected directly onto the GC column. Initial results are in ug/mL. The units are converted to total micrograms (ug) by multiplying the result (ug/mL) by the total volume (mL) contained in the impinger. See the data sheets for the reporting limits for each compound.

Receiving Notes

A Temperature Blank was included with the shipment. The temperature was measured and was not within $4 \pm 2^\circ\text{C}$. Coolant in the form of blue ice was present. Internal stability studies at Air Toxics Ltd. indicate Siloxane compounds may be stable for up to five days from collection at room temperature. Analysis proceeded.

Sample identifications on the Chain of Custody (COC) were not unique. The numbers 1 and 2 were added to each of the sample identifications to ensure uniqueness.

Analytical Notes

Impinger volumes were measured at the laboratory using a graduated cylinder and documented in the analytical logbook.

The contents of the front and back impinger for each sample were combined prior to analysis.

Sampling volume was supplied by the client. A sample volume of 21.6 L was assumed for all QC samples.

Definition of Data Qualifying Flags

Six qualifiers may have been used on the data analysis sheets and indicate as follows:

B - Compound present in laboratory blank greater than reporting limit.

J - Estimated Value.

E - Exceeds instrument calibration range.

S - Saturated peak.

Q - Exceeds quality control limits.

M - Reported value may be biased due to apparent matrix interferences.

File extensions may have been used on the data analysis sheets and indicates as follows:

a-File was requantified

b-File was quantified by a second column and detector

r1-File was requantified for the purpose of reissue

AN ENVIRONMENTAL ANALYTICAL LABORATORY

Summary of Detected Compounds SILOXANES - GC/MS

Client Sample ID: NBC-Silox #2 1/2

Lab ID#: 0712493-01AB

Compound	Rpt. Limit (ug)	Rpt. Limit (ppmv)	Amount (ug)	Amount (ppmv)
Octamethylcyclotetrasiloxane (D4)	28	0.11	140	0.52
Decamethylcyclopentasiloxane (D5)	28	0.086	3100	9.5

AN ENVIRONMENTAL ANALYTICAL LABORATORY

Client Sample ID: NBC-Silox #2 1/2

Lab ID#: 0712493-01AB

SILOXANES - GC/MS

File Name:	k122722	Date of Collection:	12/20/07
Dil. Factor:	1.00	Date of Analysis:	12/27/07 07:46 PM

Compound	Rpt. Limit (ug)	Rpt. Limit (ppmv)	Amount (ug)	Amount (ppmv)
Octamethylcyclotetrasiloxane (D4)	28	0.11	140	0.52
Decamethylcyclopentasiloxane (D5)	28	0.086	3100	9.5
Dodecamethylcyclohexasiloxane (D6)	56	0.14	Not Detected	Not Detected
Hexamethyldisiloxane	28	0.20	Not Detected	Not Detected
Octamethyltrisiloxane	28	0.13	Not Detected	Not Detected

Air Sample Volume(L): 21.6

Impinger Total Volume(mL): 28.2

Container Type: Vial

Surrogates	%Recovery	Method Limits
Hexamethyl disiloxane -d18	103	70-130

AN ENVIRONMENTAL ANALYTICAL LABORATORY

Client Sample ID: Lab Blank

Lab ID#: 0712493-02A

SILOXANES - GC/MS

File Name:	k122707	Date of Collection: NA
Dil. Factor:	1.00	Date of Analysis: 12/27/07 01:45 PM

Compound	Rpt. Limit (ug)	Rpt. Limit (ppmv)	Amount (ug)	Amount (ppmv)
Octamethylcyclotetrasiloxane (D4)	1.0	0.0038	Not Detected	Not Detected
Decamethylcyclopentasiloxane (D5)	1.0	0.0030	Not Detected	Not Detected
Dodecamethylcyclohexasiloxane (D6)	2.0	0.0051	Not Detected	Not Detected
Hexamethyldisiloxane	1.0	0.0070	Not Detected	Not Detected
Octamethyltrisiloxane	1.0	0.0048	Not Detected	Not Detected

Air Sample Volume(L): 21.6

Impinger Total Volume(mL): 1.00

Container Type: NA - Not Applicable

Surrogates	%Recovery	Method Limits
Hexamethyl disiloxane -d18	103	70-130

ESS Laboratory Certificate of Analysis

Sample Date 1/25/2008

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

PROJECT NARRATIVE

Barry Wenskowicz
Narragansett Bay Commission
2 Ernest Street
Providence, RI 02905

RE: BP Biogas
ESS Laboratory Work Order Number: 0801325

This signed Certificate of Analysis is our approved release of your analytical results. These results are only representative of sample aliquots received at the laboratory. ESS Laboratory expects its clients to follow all regulatory sampling guidelines. Beginning with this Project Narrative, the entire report has been paginated. The ESS Laboratory Certifications sheet is the final report page. This report should not be copied except in full without the approval of the laboratory. Samples will be disposed of thirty days after the final report has been mailed. If you have any questions or concerns, please feel free to call our Customer Service Department.

Laurel Stoddard
Laboratory Director

Date: February 06, 2008

Analytical Summary

The project as described above has been analyzed in accordance with the ESS Quality Assurance Plan. This plan utilizes the following methodologies: US EPA SW-846, US EPA Methods for Chemical Analysis of Water and Wastes per 40 CFR Part 136, APHA Standard Methods for the Examination of Water and Wastewater, American Society for Testing and Materials (ASTM), and other recognized methodologies. The analyses with these noted observations are in conformance to the Quality Assurance Plan. In chromatographic analysis, manual integration may be used instead of automated integration because it produces more accurate results. All ICP Metals were analyzed using the established linear dynamic range to determine acceptable analytical results.

ESS Laboratory certifies that the test results meet the requirements of NELAC, except where noted within this project narrative.

Samples were analyzed in accordance with the Guidelines Establishing Test Procedures for the Analysis of Pollutants, 40 CFR Part 136, as amended.

Sample Receipt

The following sample(s) were received on January 25, 2008 for the analyses specified on the enclosed Chain of Custody Record.

Laboratory ID	Matrix	Client Sample ID
0801325-01	Aqueous	Front Raw
0801325-02	Aqueous	Front Chilled

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0801325

PROJECT NARRATIVE

Classical Chemistry

0801325-01 **Silicon Breakthrough 1.6%**
 Silicon
0801325-02 **Silicon Breakthrough 0.8%**
 Silicon

Siloxane Analysis

0801325-01 **D5 Breakthrough = 1.2%**
 Decamethylcyclopentasiloxane (D5)
0801325-01 **No D4 Breakthrough Detected**
 Octamethylcyclotetrasiloxane (D4)
0801325-02 **D5 Breakthrough = 0.5%**
 Decamethylcyclopentasiloxane (D5)
0801325-02 **No D4 Breakthrough Detected**
 Octamethylcyclotetrasiloxane (D4)

No other observations noted.

End of Project Narrative.

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front Raw
Date Sampled: 01/25/08 15:00
Percent Solids: N/A

ESS Laboratory Work Order: 0801325
ESS Laboratory Sample ID: 0801325-01
Sample Matrix: Aqueous

Classical Chemistry

<u>Analyte</u>		<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	4a	2520	ug/sample	1700	3111D		20	AA	02/01/08

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front Raw
Date Sampled: 01/25/08 15:00
Percent Solids: N/A
Initial Volume: 1
Final Volume: 17.03
Extraction Method: 3580A

ESS Laboratory Work Order: 0801325
ESS Laboratory Sample ID: 0801325-01
Sample Matrix: Aqueous
Analyst: VSC
Prepared: 01/31/08

Siloxane Analysis

Analyte		Results	Units	MRI	Method	Limit	DF	Analyst	Analyzed	I/V	F/Y
Decamethylcyclopentasiloxane (D5)	4	3680	ug/sample	34.1	8270C		10	VSC	01/31/08	1	17.03
Octamethylcyclotetrasiloxane (D4)	6	158	ug/sample	3.41	8270C		1	VSC	01/31/08	1	17.03

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4

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front Chilled
Date Sampled: 01/25/08 15:00
Percent Solids: N/A

ESS Laboratory Work Order: 0801325
ESS Laboratory Sample ID: 0801325-02
Sample Matrix: Aqueous

Classical Chemistry

<u>Analyte</u>		<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	5a	4190	ug/sample	1650	3111D		20	AA	02/01/08

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front Chilled
Date Sampled: 01/25/08 15:00
Percent Solids: N/A
Initial Volume: 1
Final Volume: 16.46
Extraction Method: 3580A

ESS Laboratory Work Order: 0801325
ESS Laboratory Sample ID: 0801325-02
Sample Matrix: Aqueous
Analyst: VSC
Prepared: 01/31/08

Siloxane Analysis

Analyte		Results	Units	MRL	Method	Limit	DF	Analyst	Analyzed	I/V	F/V
Decamethylcyclotrasiloxane (D5)	5	5460	ug/sample	63.8	8270C		20	VSC	01/31/08	1	16.46
Octamethylcyclotetrasiloxane (D4)	6	226	ug/sample	3.29	8270C		1	VSC	01/31/08	1	16.46

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0801325

Quality Control Data

Analyte	Result	MRL	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Qualifier
Classical Chemistry										
Batch BB80108 - Metals No Prep										
Blank										
Silicon	ND	100	ug/sample							
Siloxane Analysis										
Batch BA83131 - 3580A										
Blank										
Decamethylcyclopentasiloxane (D5)	ND	4.00	ug/sample							
Octamethylcyclotetrasiloxane (D4)	ND	4.00	ug/sample

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0801325

Notes and Definitions

U	Analyte included in the analysis, but not detected
D	Diluted.
6	No D4 Breakthrough Detected
5a	Silicon Breakthrough 0.8%
5	D5 Breakthrough = 0.5%
4a	Silicon Breakthrough 1.6%
4	D5 Breakthrough = 1.2%
ND	Analyte NOT DETECTED above the detection limit
dry	Sample results reported on a dry weight basis
RPD	Relative Percent Difference
MDL	Method Detection Limit
MRL	Method Reporting Limit
I/V	Initial Volume
F/V	Final Volume
§	Subcontracted analysis; see attached report
1	Range result excludes concentrations of surrogates and/or internal standards eluting in that range.
2	Range result excludes concentrations of target analytes eluting in that range.
3	Range result excludes the concentration of the C9-C10 aromatic range.
Avg	Results reported as a mathematical average.

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0801325

ESS LABORATORY CERTIFICATIONS

U.S. Army Corps of Engineers
Soil and Water

Navy Installation Restoration QA Program
Soil and Water

Rhode Island: A-179

Connecticut: PH-0750

Maine: RI002

Massachusetts: M-RI002

New Hampshire (NELAP accredited): 242405
Potable Water
Non Potable Water

New York (NELAP accredited): 11313
Potable Water
Non Potable Water
Solid and Hazardous Waste

United States Department of Agriculture
Soil Permit: S-54210

New Jersey (NELAP accredited): RI002
Potable Water
Non Potable Water
Soil and Hazardous Waste

Maryland: 301
Potable Water

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9

EMSL Analytical Certificate of Analysis

Sample Date 6/16/2008

EMSL Analytical, Inc.

107 Haddon Avenue, Westmont, NJ 08108
Phone: (856) 858-4800

Materials Science Division

Attn.: Barry Wenskowicz
Narragansett Bay Commission
One Service Road
Providence, RI 02905

Phone: 401-461-8848 Fax: 401-461-6540

EMSL Case No.: 360800841 revised
Sample(s) Received: 06/17/08
Date of Analysis: 07/08/08
Date Printed: 07/18/08
Reported By: Eric Barrett

**- Laboratory Report -
Thermal Conductivity
For
Project: Silica Conductivity**

Analyzed by:

Eric W. Barrett, Ph.D.
Polymer Chemist

07/09/2008

Date

QA/QC:

John Newton
Laboratory Manager

07/09/2008

Date

EMSL**EMSL Analytical, Inc.**107 Haddon Avenue, Westmont, NJ 08108
Phone: (856) 858-4800Attn.: *Barry Wenskowicz*
Narragansett Bay Commission
One Service Road
Providence, RI 02905

Phone: 401-461-8848 Fax: 401-461-6540

EMSL Case No.: 360800841 revised
Sample(s) Received: 06/17/08
Date of Analysis: 07/08/08
Date Printed: 07/18/08
Reported By: Eric Barrett*Procurement of Samples and Analytical Overview:*

The samples for analysis arrived at EMSL Analytical's corporate laboratory in Westmont, NJ on 06/17/2008. The package arrived in satisfactory condition with no evidence of damage to the contents. The purpose of the analysis is to determine the thermal conductivity. The data reported herein has been obtained using the following equipment and methodologies.

Methods & Equipment: Thermal analysis

EMSL *EMSL Analytical, Inc.*

107 Haddon Avenue, Westmont, NJ 08108
Phone: (856) 858-4800

Attn.: *Barry Wenskowicz*
Narragansett Bay Commission
One Service Road
Providence, RI 02905

Phone: 401-461-8848 Fax: 401-461-6540

EMSL Case No.: 360800841 revised
Sample(s) Received: 06/17/08
Date of Analysis: 07/08/08
Date Printed: 07/18/08
Reported By: Eric Barrett

Results and Discussion:

Sample	Description	dT	Thermal Conductivity
1	Boiler Silica Composite/Hex Bldg	100°C	0.049 W/m°C
		300°C	0.056 W/m°C

Note: Equipment calibrated against vermiculite (0.058 W/m°C)

The results are obtained using the methods and sampling procedures as described in the report or as stated in the published standard methods, and are only guaranteed to the accuracy and precision consistent with the used methods and sampling procedures. Any change in methods and sampling procedure may generate substantially different results. EMSL Analytical, Inc. assumes no responsibility or liability for the manner in which the results are used or interpreted.

Title

Standard Practice for Calculating Thermal Transmission Properties Under Steady- State Conditions

ASTM International

Publication Date:

Nov 1, 2007

1. Scope

1.1 This practice provides the user with a uniform procedure for calculating the thermal transmission properties of a material or system from data generated by steady state, one dimensional test methods used to determine heat flux and surface temperatures. This practice is intended to eliminate the need for similar calculation sections in Test Methods C 177, C 335, C 518, C 976, C 1033, C 1114 and C 1363 by permitting use of these standard calculation forms by reference.

1.2 The thermal transmission properties described include: thermal conductance, thermal resistance, apparent thermal conductivity, apparent thermal resistivity, surface conductance, surface resistance, and overall thermal resistance or transmittance.

1.3 This practice provides the method for developing the apparent thermal conductivity as a function of temperature relationship for a specimen from data generated by standard test methods at small or large temperature differences. This relationship can be used to characterize material for comparison to material specifications and for use in calculation programs such as Practice C 680.

1.4 The SI unit values used in this practice are considered standard.

1.5 This practice includes a discussion of the definitions and underlying assumptions for the calculation of thermal transmission properties. Tests to detect deviations from these assumptions are described. This practice also considers the complicating effects of uncertainties due to the measurement processes and material variability. See Section 7.

1.6 This practice is not intended to cover all possible aspects of thermal properties data base development. For new materials, the user should investigate the variations in thermal properties seen in similar materials. The information contained in Section 7, the Appendix and the technical papers listed in the References section of this practice may be helpful in determining whether the material under study has thermal properties that can be described by equations using this practice. Some examples where this method has limited application include: (1) the onset of convection in insulation as described in Reference (21); (2) a phase change of one of the insulation system components such as a blowing gas in foam; and (3) the influence of heat flow direction and temperature difference changes for reflective insulations.

ESS Laboratory Certificate of Analysis

Sample Date 6/20/2008

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

PROJECT NARRATIVE

Barry Wenskowicz
Narragansett Bay Commission
2 Ernest Street
Providence, RI 02905

RE: BP Biogas
ESS Laboratory Work Order Number: 0806315

This signed Certificate of Analysis is our approved release of your analytical results. These results are only representative of sample aliquots received at the laboratory. ESS Laboratory expects its clients to follow all regulatory sampling guidelines. Beginning with this Project Narrative, the entire report has been paginated. The ESS Laboratory Certifications sheet is the final report page. This report should not be copied except in full without the approval of the laboratory. Samples will be disposed of thirty days after the final report has been mailed. If you have any questions or concerns, please feel free to call our Customer Service Department.

Laurel Stoddard
Laboratory Director

Date: June 27, 2008

Analytical Summary

The project as described above has been analyzed in accordance with the ESS Quality Assurance Plan. This plan utilizes the following methodologies: US EPA SW-846, US EPA Methods for Chemical Analysis of Water and Wastes per 40 CFR Part 136, APHA Standard Methods for the Examination of Water and Wastewater, American Society for Testing and Materials (ASTM), and other recognized methodologies. The analyses with these noted observations are in conformance to the Quality Assurance Plan. In chromatographic analysis, manual integration may be used instead of automated integration because it produces more accurate results. All ICP Metals were analyzed using the established linear dynamic range to determine acceptable analytical results.

ESS Laboratory certifies that the test results meet the requirements of NELAC, except where noted within this project narrative.

Samples were analyzed in accordance with the Guidelines Establishing Test Procedures for the Analysis of Pollutants, 40 CFR Part 136, as amended.

Sample Receipt

The following sample(s) were received on June 20, 2008 for the analyses specified on the enclosed Chain of Custody Record.

Laboratory ID	Matrix	Client Sample ID
0806315-01	Aqueous	Front Raw
0806315-02	Aqueous	Back Raw
0806315-03	Aqueous	Front Pre-Chill
0806315-04	Aqueous	Back Pre-Chill

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Fax: 401-461-4486

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ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0806315

PROJECT NARRATIVE

No unusual observations noted.

End of Project Narrative.

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CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0806315

Classical Chemistry

Client Sample ID: Front Raw
Date Sampled: 06/20/08 13:00
Percent Solids: N/A

ESS Laboratory Sample ID: 0806315-01
Sample Matrix: Aqueous

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	12800	ug/sample	1710	3111D	20	JP	06/21/08

Client Sample ID: Back Raw
Date Sampled: 06/20/08 13:00
Percent Solids: N/A

ESS Laboratory Sample ID: 0806315-02
Sample Matrix: Aqueous

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	361	ug/sample	93	3111D	1	JP	06/21/08

Client Sample ID: Front Pre-Chill
Date Sampled: 06/20/08 13:00
Percent Solids: N/A

ESS Laboratory Sample ID: 0806315-03
Sample Matrix: Aqueous

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	3360	ug/sample	846	3111D	10	JP	06/21/08

Client Sample ID: Back Pre-Chill
Date Sampled: 06/20/08 13:00
Percent Solids: N/A

ESS Laboratory Sample ID: 0806315-04
Sample Matrix: Aqueous

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	193	ug/sample	94	3111D	1	JP	06/21/08

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0806315

Quality Control Data

Analyte	Result	MRL	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Qualifier
Classical Chemistry										
Batch BF82102 - Metals No Prep										
Duplicate	Source: 0806315-04									
Silicon	191	93.6	ug/sample		193			0.8	20	

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CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0806315

Notes and Definitions

D	Diluted.
ND	Analyte NOT DETECTED above the detection limit
dry	Sample results reported on a dry weight basis
RPD	Relative Percent Difference
MDL	Method Detection Limit
MRL	Method Reporting Limit
I/V	Initial Volume
F/V	Final Volume
§	Subcontracted analysis; see attached report
1	Range result excludes concentrations of surrogates and/or internal standards eluting in that range.
2	Range result excludes concentrations of target analytes eluting in that range.
3	Range result excludes the concentration of the C9-C10 aromatic range.
Avg	Results reported as a mathematical average.

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Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0806315

ESS LABORATORY CERTIFICATIONS

U.S. Army Corps of Engineers
Soil and Water

Navy Installation Restoration QA Program
Soil and Water

Rhode Island: A-179

Connecticut: PH-0750

Maine: RI002

Massachusetts: M-RI002

New Hampshire (NELAP accredited): 242405
Potable Water
Non Potable Water

New York (NELAP accredited): 11313
Potable Water
Non Potable Water
Solid and Hazardous Waste

United States Department of Agriculture
Soil Permit: S-54210

New Jersey (NELAP accredited): RI002
Potable Water
Non Potable Water
Soil and Hazardous Waste

Maryland: 301
Potable Water

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ESS Laboratory Certificate of Analysis

Sample Date 8/6/2008

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

PROJECT NARRATIVE

Barry Wenskowicz
Narragansett Bay Commission
One Service Road
Providence, RI 02905

RE: BP Biogas
ESS Laboratory Work Order Number: 0808085

This signed Certificate of Analysis is our approved release of your analytical results. These results are only representative of sample aliquots received at the laboratory. ESS Laboratory expects its clients to follow all regulatory sampling guidelines. Beginning with this Project Narrative, the entire report has been paginated. The ESS Laboratory Certifications sheet is the final report page. This report should not be copied except in full without the approval of the laboratory. Samples will be disposed of thirty days after the final report has been mailed. If you have any questions or concerns, please feel free to call our Customer Service Department.

Laurel Stoddard
Laboratory Director

Date: August 25, 2008

Analytical Summary

The project as described above has been analyzed in accordance with the ESS Quality Assurance Plan. This plan utilizes the following methodologies: US EPA SW-846, US EPA Methods for Chemical Analysis of Water and Wastes per 40 CFR Part 136, APHA Standard Methods for the Examination of Water and Wastewater, American Society for Testing and Materials (ASTM), and other recognized methodologies. The analyses with these noted observations are in conformance to the Quality Assurance Plan. In chromatographic analysis, manual integration may be used instead of automated integration because it produces more accurate results. All ICP Metals were analyzed using the established linear dynamic range to determine acceptable analytical results.

ESS Laboratory certifies that the test results meet the requirements of NELAC, except where noted within this project narrative.

Samples were analyzed in accordance with the Guidelines Establishing Test Procedures for the Analysis of Pollutants, 40 CFR Part 136, as amended.

Sample Receipt

The following sample(s) were received on August 06, 2008 for the analyses specified on the enclosed Chain of Custody Record.

GC/MS Scan: Additional peaks in the chromatogram were evaluated to determine if they were compounds containing silicon. Our library search did not identify any additional compounds with silicon.

Laboratory ID
0808085-01
0808085-02

Matrix
Aqueous
Aqueous

Client Sample ID
Pre-Chill Front
Pre-Chill Back

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ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0808085

PROJECT NARRATIVE

No unusual observations noted.

End of Project Narrative.

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ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Pre-Chill Front
Date Sampled: 08/06/08 14:00
Percent Solids: N/A
Initial Volume: 1
Final Volume: 16.83
Extraction Method: No Prep

ESS Laboratory Work Order: 0808085
ESS Laboratory Sample ID: 0808085-01
Sample Matrix: Aqueous
Analyst: VSC
Prepared: 08/07/08

Siloxane Analysis

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>	<u>L/V</u>	<u>F/V</u>
Decamethylcyclopentasiloxane (D5)	6220	ug/Sample	67	GC/MS		4	VSC	08/08/08	1	16.83
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	17	GC/MS		1	VSC	08/07/08	1	16.83
Hexamethyldisiloxane (MM)	ND	ug/Sample	17	GC/MS		1	VSC	08/07/08	1	16.83
Octamethylcyclotetrasiloxane (D4)	2820	ug/Sample	67	GC/MS		4	VSC	08/08/08	1	16.83

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Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Pre-Chill Back
Date Sampled: 08/06/08 14:00
Percent Solids: N/A
Initial Volume: 1
Final Volume: 18.87
Extraction Method: No Prep

ESS Laboratory Work Order: 0808085
ESS Laboratory Sample ID: 0808085-02
Sample Matrix: Aqueous
Analyst: VSC
Prepared: 08/07/08

Siloxane Analysis

Analyte	Results	Units	MRL	Method	Limit	DF	Analyst	Analyzed	I/V	F/V
Decamethylcyclopentasiloxane (D5)	68	ug/Sample	19	GC/MS		1	VSC	08/07/08	1	18.87
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	19	GC/MS		1	VSC	08/07/08	1	18.87
Hexamethyldisiloxane (MM)	ND	ug/Sample	19	GC/MS		1	VSC	08/07/08	1	18.87
Octamethylcyclotetrasiloxane (D4)	55	ug/Sample	19	GC/MS		1	VSC	08/07/08	1	18.87

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Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0808085

Quality Control Data

Analyte	Result	MRL	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Qualifier
Siloxane Analysis										
Batch BH80729 - No Prep										
Blank										
Decamethylcyclopentasiloxane (D5)	ND	20	ug/Sample							
Hexamethylcyclotrisiloxane (D3)	ND	20	ug/Sample							
Hexamethyldisiloxane (MM)	ND	20	ug/Sample							
Octamethylcyclotetrasiloxane (D4)	ND	20	ug/Sample							
LCS										
Decamethylcyclopentasiloxane (D5)	980	20	ug/Sample	1000		98	70-130			
Hexamethylcyclotrisiloxane (D3)	996	20	ug/Sample	1000		100	70-130			
Hexamethyldisiloxane (MM)	975	20	ug/Sample	999.6		98	70-130			
Octamethylcyclotetrasiloxane (D4)	1010	20	ug/Sample	999.6		101	70-130			
Duplicate Source: 0808085-01										
Decamethylcyclopentasiloxane (D5)	6860	73	ug/Sample		6220			10	20	
Hexamethylcyclotrisiloxane (D3)	ND	18	ug/Sample		ND				20	
Hexamethyldisiloxane (MM)	ND	18	ug/Sample		ND				20	
Octamethylcyclotetrasiloxane (D4)	3160	73	ug/Sample		2820			11	20	

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Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0808085

Notes and Definitions

U	Analyte included in the analysis, but not detected
D	Diluted.
ND	Analyte NOT DETECTED above the detection limit
dry	Sample results reported on a dry weight basis
RPD	Relative Percent Difference
MDL	Method Detection Limit
MRL	Method Reporting Limit
I/V	Initial Volume
F/V	Final Volume
§	Subcontracted analysis; see attached report
1	Range result excludes concentrations of surrogates and/or internal standards eluting in that range.
2	Range result excludes concentrations of target analytes eluting in that range.
3	Range result excludes the concentration of the C9-C10 aromatic range.
Avg	Results reported as a mathematical average.

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Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0808085

ESS LABORATORY CERTIFICATIONS

U.S. Army Corps of Engineers
Soil and Water

Navy Installation Restoration QA Program
Soil and Water

Rhode Island: A-179

Connecticut: PH-0750

Maine: RI002

Massachusetts: M-RI002

New Hampshire (NELAP accredited): 242405
Potable Water
Non Potable Water

New York (NELAP accredited): 11313
Potable Water
Non Potable Water
Solid and Hazardous Waste

United States Department of Agriculture
Soil Permit: S-54210

New Jersey (NELAP accredited): RI002
Potable Water
Non Potable Water
Soil and Hazardous Waste

Maryland: 301
Potable Water

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ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

PROJECT NARRATIVE

Barry Wenskowicz
Narragansett Bay Commission
One Service Road
Providence, RI 02905

RE: BP Biogas
ESS Laboratory Work Order Number: 0808086

This signed Certificate of Analysis is our approved release of your analytical results. These results are only representative of sample aliquots received at the laboratory. ESS Laboratory expects its clients to follow all regulatory sampling guidelines. Beginning with this Project Narrative, the entire report has been paginated. The ESS Laboratory Certifications sheet is the final report page. This report should not be copied except in full without the approval of the laboratory. Samples will be disposed of thirty days after the final report has been mailed. If you have any questions or concerns, please feel free to call our Customer Service Department.

Laurel Stoddard
Laboratory Director

Date: August 25, 2008

Analytical Summary

The project as described above has been analyzed in accordance with the ESS Quality Assurance Plan. This plan utilizes the following methodologies: US EPA SW-846, US EPA Methods for Chemical Analysis of Water and Wastes per 40 CFR Part 136, APHA Standard Methods for the Examination of Water and Wastewater, American Society for Testing and Materials (ASTM), and other recognized methodologies. The analyses with these noted observations are in conformance to the Quality Assurance Plan. In chromatographic analysis, manual integration may be used instead of automated integration because it produces more accurate results. All ICP Metals were analyzed using the established linear dynamic range to determine acceptable analytical results.

ESS Laboratory certifies that the test results meet the requirements of NELAC, except where noted within this project narrative.

Samples were analyzed in accordance with the Guidelines Establishing Test Procedures for the Analysis of Pollutants, 40 CFR Part 136, as amended.

Sample Receipt

The following sample(s) were received on August 06, 2008 for the analyses specified on the enclosed Chain of Custody Record.

GC/MS Scan: Additional peaks in the chromatogram were evaluated to determine if they were compounds containing silicon. Our library search revealed a compound at 7.01 minutes and identified it as Dodecamethylcyclohexasiloxane (CAS# 540-97-6) with an estimated concentration of 445 ug/sample.

Laboratory ID
0808086-01
0808086-02

Matrix
Aqueous
Aqueous

Client Sample ID
Raw Front
Raw Back

D6

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ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0808086

PROJECT NARRATIVE

No unusual observations noted.

End of Project Narrative.

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ESS Laboratory

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CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Raw Front
Date Sampled: 08/06/08 14:00
Percent Solids: N/A
Initial Volume: 1
Final Volume: 18.29
Extraction Method: No Prep

ESS Laboratory Work Order: 0808086
ESS Laboratory Sample ID: 0808086-01
Sample Matrix: Aqueous
Analyst: VSC
Prepared: 08/07/08

Siloxane Analysis

Analyte	Results	Units	MRL	Method	Limit	DF	Analyst	Analyzed	I/V	F/V
Decamethylcyclotrasiloxane (D5)	25000	ug/Sample	183	GC/MS		10	VSC	08/08/08	1	18.29
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	18	GC/MS		1	VSC	08/07/08	1	18.29
Hexamethyldisiloxane (MM)	ND	ug/Sample	18	GC/MS		1	VSC	08/07/08	1	18.29
Octamethylcyclotetrasiloxane (D4)	3530	ug/Sample	183	GC/MS		10	VSC	08/08/08	1	18.29

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ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Raw Back
Date Sampled: 08/06/08 14:00
Percent Solids: N/A
Initial Volume: 1
Final Volume: 18.84
Extraction Method: No Prep

ESS Laboratory Work Order: 0808086
ESS Laboratory Sample ID: 0808086-02
Sample Matrix: Aqueous
Analyst: VSC
Prepared: 08/07/08

Siloxane Analysis

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>	<u>I/V</u>	<u>F/V</u>
Decamethylcyclopentasiloxane (D5)	291	ug/Sample	19	GC/MS		1	VSC	08/07/08	1	18.84
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	19	GC/MS		1	VSC	08/07/08	1	18.84
Hexamethyldisiloxane (MM)	ND	ug/Sample	19	GC/MS		1	VSC	08/07/08	1	18.84
Octamethylcyclotetrasiloxane (D4)	87	ug/Sample	19	GC/MS		1	VSC	08/07/08	1	18.84

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Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0808086

Quality Control Data

Analyte	Result	MRL	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Qualifier
Siloxane Analysis										
Batch BH80729 - No Prep										
Blank										
Decamethylcyclopentasiloxane (D5)	ND	20	ug/Sample							
Hexamethylcyclotrisiloxane (D3)	ND	20	ug/Sample							
Hexamethyldisiloxane (MM)	ND	20	ug/Sample							
Octamethylcyclotetrasiloxane (D4)	ND	20	ug/Sample							
LCS										
Decamethylcyclopentasiloxane (D5)	980	20	ug/Sample	1000		98	70-130			
Hexamethylcyclotrisiloxane (D3)	996	20	ug/Sample	1000		100	70-130			
Hexamethyldisiloxane (MM)	975	20	ug/Sample	999.6		98	70-130			
Octamethylcyclotetrasiloxane (D4)	1010	20	ug/Sample	999.6		101	70-130			

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ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0808086

Notes and Definitions

U	Analyte included in the analysis, but not detected
D	Diluted.
ND	Analyte NOT DETECTED above the detection limit
dry	Sample results reported on a dry weight basis
RPD	Relative Percent Difference
MDL	Method Detection Limit
MRL	Method Reporting Limit
I/V	Initial Volume
F/V	Final Volume
§	Subcontracted analysis; see attached report
1	Range result excludes concentrations of surrogates and/or internal standards eluting in that range.
2	Range result excludes concentrations of target analytes eluting in that range.
3	Range result excludes the concentration of the C9-C10 aromatic range.
Avg	Results reported as a mathematical average.

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Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0808086

ESS LABORATORY CERTIFICATIONS

U.S. Army Corps of Engineers
Soil and Water

Navy Installation Restoration QA Program
Soil and Water

Rhode Island: A-179

Connecticut: PH-0750

Maine: RI002

Massachusetts: M-RI002

New Hampshire (NELAP accredited): 242405
Potable Water
Non Potable Water

New York (NELAP accredited): 11313
Potable Water
Non Potable Water
Solid and Hazardous Waste

United States Department of Agriculture
Soil Permit: S-54210

New Jersey (NELAP accredited): RI002
Potable Water
Non Potable Water
Soil and Hazardous Waste

Maryland: 301
Potable Water

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CERTIFICATE OF ANALYSIS

PROJECT NARRATIVE

Barry Wenskowicz
Narragansett Bay Commission
One Service Road
Providence, RI 02905

RE: BP Biogas
ESS Laboratory Work Order Number: 0809088

This signed Certificate of Analysis is our approved release of your analytical results. These results are only representative of sample aliquots received at the laboratory. ESS Laboratory expects its clients to follow all regulatory sampling guidelines. Beginning with this Project Narrative, the entire report has been paginated. The ESS Laboratory Certifications sheet is the final report page. This report should not be copied except in full without the approval of the laboratory. Samples will be disposed of thirty days after the final report has been mailed. If you have any questions or concerns, please feel free to call our Customer Service Department.

Laurel Stoddard
Laboratory Director

Date: September 10, 2008

Analytical Summary

The project as described above has been analyzed in accordance with the ESS Quality Assurance Plan. This plan utilizes the following methodologies: US EPA SW-846, US EPA Methods for Chemical Analysis of Water and Wastes per 40 CFR Part 136, APHA Standard Methods for the Examination of Water and Wastewater, American Society for Testing and Materials (ASTM), and other recognized methodologies. The analyses with these noted observations are in conformance to the Quality Assurance Plan. In chromatographic analysis, manual integration may be used instead of automated integration because it produces more accurate results. All ICP Metals were analyzed using the established linear dynamic range to determine acceptable analytical results.

ESS Laboratory certifies that the test results meet the requirements of NELAC, except where noted within this project narrative.

Samples were analyzed in accordance with the Guidelines Establishing Test Procedures for the Analysis of Pollutants, 40 CFR Part 136, as amended.

Sample Receipt

The following sample(s) were received on September 08, 2008 for the analyses specified on the enclosed Chain of Custody Record.

Laboratory ID	Matrix	Client Sample ID
0809088-01	Aqueous	Pre-Chill Front
0809088-02	Aqueous	Pre-Chill Back
0809088-03	Aqueous	Raw Front
0809088-04	Aqueous	Raw Back

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CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0809088

PROJECT NARRATIVE

No unusual observations noted.

End of Project Narrative.

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CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0809088

Classical Chemistry

Client Sample ID: Pre-Chill Front
Date Sampled: 08/06/08 14:00
Percent Solids: N/A

ESS Laboratory Sample ID: 0809088-01
Sample Matrix: Aqueous

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	4920	ug/sample	1430	3111D	20	JP	09/09/08

Client Sample ID: Pre-Chill Back
Date Sampled: 08/06/08 14:00
Percent Solids: N/A

ESS Laboratory Sample ID: 0809088-02
Sample Matrix: Aqueous

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	ND	ug/sample	86	3111D	1	JP	09/09/08

Client Sample ID: Raw Front
Date Sampled: 08/06/08 14:00
Percent Solids: N/A

ESS Laboratory Sample ID: 0809088-03
Sample Matrix: Aqueous

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	15500	ug/sample	4180	3111D	50	JP	09/09/08

Client Sample ID: Raw Back
Date Sampled: 08/06/08 14:00
Percent Solids: N/A

ESS Laboratory Sample ID: 0809088-04
Sample Matrix: Aqueous

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	230	ug/sample	90	3111D	1	JP	09/09/08

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CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0809088

Quality Control Data

Analyte	Result	MRL	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Qualifier
Classical Chemistry										
Batch B180901 - Metals No Prep										
Duplicate Source: 0809088-04										
Silicon	205	90.4	ug/sample		230			11	20	

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CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0809088

Notes and Definitions

U	Analyte included in the analysis, but not detected
D	Diluted.
ND	Analyte NOT DETECTED above the detection limit
dry	Sample results reported on a dry weight basis
RPD	Relative Percent Difference
MDL	Method Detection Limit
MRL	Method Reporting Limit
I/V	Initial Volume
F/V	Final Volume
§	Subcontracted analysis; see attached report
1	Range result excludes concentrations of surrogates and/or internal standards eluting in that range.
2	Range result excludes concentrations of target analytes eluting in that range.
3	Range result excludes the concentration of the C9-C10 aromatic range.
Avg	Results reported as a mathematical average.

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CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0809088

ESS LABORATORY CERTIFICATIONS

U.S. Army Corps of Engineers
Soil and Water

Navy Installation Restoration QA Program
Soil and Water

Rhode Island: A-179

Connecticut: PH-0750

Maine: RI002

Massachusetts: M-RI002

New Hampshire (NELAP accredited): 242405
Potable Water
Non Potable Water

New York (NELAP accredited): 11313
Potable Water
Non Potable Water
Solid and Hazardous Waste

United States Department of Agriculture
Soil Permit: S-54210

New Jersey (NELAP accredited): RI002
Potable Water
Non Potable Water
Soil and Hazardous Waste

Maryland: 301
Potable Water

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ESS Laboratory Certificate of Analysis

Sample Date 12/23/2008

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

PROJECT NARRATIVE

Barry Wenskowicz
Narragansett Bay Commission
One Service Road
Providence, RI 02905

RE: BP Biogas

ESS Laboratory Work Order Number: 0812357

This signed Certificate of Analysis is our approved release of your analytical results. These results are only representative of sample aliquots received at the laboratory. ESS Laboratory expects its clients to follow all regulatory sampling guidelines. Beginning with this Project Narrative, the entire report has been paginated. The ESS Laboratory Certifications sheet is the final report page. This report should not be copied except in full without the approval of the laboratory. Samples will be disposed of thirty days after the final report has been delivered. If you have any questions or concerns, please feel free to call our Customer Service Department.

Laurel Stoddard
Laboratory Director

Date: January 13, 2009

Analytical Summary

The project as described above has been analyzed in accordance with the ESS Quality Assurance Plan. This plan utilizes the following methodologies: US EPA SW-846, US EPA Methods for Chemical Analysis of Water and Wastes per 40 CFR Part 136, APHA Standard Methods for the Examination of Water and Wastewater, American Society for Testing and Materials (ASTM), and other recognized methodologies. The analyses with these noted observations are in conformance to the Quality Assurance Plan. In chromatographic analysis, manual integration may be used instead of automated integration because it produces more accurate results. All ICP Metals were analyzed using the established linear dynamic range to determine acceptable analytical results.

ESS Laboratory certifies that the test results meet the requirements of NELAC, except where noted within this project narrative.

Samples were analyzed in accordance with the Guidelines Establishing Test Procedures for the Analysis of Pollutants, 40 CFR Part 136, as amended.

Sample Receipt

The following sample(s) were received on December 23, 2008 for the analyses specified on the enclosed Chain of Custody Record.

Laboratory ID
0812357-01
0812357-02

Matrix
Aqueous
Aqueous

Client Sample ID
NBC BP Biogas Front
NBC BP Biogas Back

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0812357

PROJECT NARRATIVE

No unusual observations noted.

End of Project Narrative.

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: NBC BP Biogas Front
Date Sampled: 12/23/08 14:10
Percent Solids: N/A

ESS Laboratory Work Order: 0812357
ESS Laboratory Sample ID: 0812357-01
Sample Matrix: Aqueous

Classical Chemistry

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	2490	ug/sample	1000	3111D		10	JP	01/07/09

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: NBC BP Biogas Front
Date Sampled: 12/23/08 14:10
Percent Solids: N/A
Initial Volume: 1
Final Volume: 20
Extraction Method: No Prep

ESS Laboratory Work Order: 0812357
ESS Laboratory Sample ID: 0812357-01
Sample Matrix: Aqueous
Analyst: VSC
Prepared: 12/29/08

Siloxane Analysis

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>	<u>I/V</u>	<u>F/V</u>
Decamethylcyclopentasiloxane (D5)	6100	ug/Sample	800	GC/MS		4	VSC	01/09/09	1	20
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	200	GC/MS		1	VSC	01/09/09	1	20
Hexamethyldisiloxane (MM)	ND	ug/Sample	200	GC/MS		1	VSC	01/09/09	1	20
Octamethylcyclotetrasiloxane (D4)	233	ug/Sample	200	GC/MS		1	VSC	01/09/09	1	20

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CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: NBC BP Biogas Back
Date Sampled: 12/23/08 14:10
Percent Solids: N/A

ESS Laboratory Work Order: 0812357
ESS Laboratory Sample ID: 0812357-02
Sample Matrix: Aqueous

Classical Chemistry

Analyte
Silicon

Results
ND

Units
ug/sample

MRL
100

Method
3111D

Limit

DF
1

Analyst
JP

Analyzed
01/07/09

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Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: NBC BP Biogas Back
Date Sampled: 12/23/08 14:10
Percent Solids: N/A
Initial Volume: 1
Final Volume: 20
Extraction Method: No Prep

ESS Laboratory Work Order: 0812357
ESS Laboratory Sample ID: 0812357-02
Sample Matrix: Aqueous
Analyst: VSC
Prepared: 12/29/08

Siloxane Analysis

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>	<u>I/V</u>	<u>F/V</u>
Decamethylcyclopentasiloxane (D5)	ND	ug/Sample	200	GC/MS		1	VSC	01/09/09	1	20
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	200	GC/MS		1	VSC	01/09/09	1	20
Hexamethyldisiloxane (MM)	ND	ug/Sample	200	GC/MS		1	VSC	01/09/09	1	20
Octamethylcyclotetrasiloxane (D4)	ND	ug/Sample	200	GC/MS		1	VSC	01/09/09	1	20

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0812357

Quality Control Data

Analyte	Result	MRL	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Qualifier
Classical Chemistry										
Batch BA90601 - Metals No Prep										
Duplicate	Source: 0812357-02									
Silicon	ND	100	ug/sample		ND				20	
Siloxane Analysis										
Batch BL82926 - No Prep										
Blank										
Decamethylcyclopentasiloxane (D5)	ND	200	ug/Sample							
Hexamethylcyclotrisiloxane (D3)	ND	200	ug/Sample							
Hexamethyldisiloxane (MM)	ND	200	ug/Sample							
Octamethylcyclotetrasiloxane (D4)	ND	200	ug/Sample							
Duplicate	Source: 0812357-01									
Decamethylcyclopentasiloxane (D5)	6110	800	ug/Sample		6100			0.2	20	
Hexamethylcyclotrisiloxane (D3)	ND	200	ug/Sample		ND				20	
Hexamethyldisiloxane (MM)	ND	200	ug/Sample		ND				20	
Octamethylcyclotetrasiloxane (D4)	239	200	ug/Sample		233			3	20	

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Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0812357

Notes and Definitions

U	Analyte included in the analysis, but not detected
D	Diluted.
ND	Analyte NOT DETECTED above the detection limit
dry	Sample results reported on a dry weight basis
RPD	Relative Percent Difference
MDL	Method Detection Limit
MRL	Method Reporting Limit
I/V	Initial Volume
F/V	Final Volume
§	Subcontracted analysis; see attached report
1	Range result excludes concentrations of surrogates and/or internal standards eluting in that range.
2	Range result excludes concentrations of target analytes eluting in that range.
3	Range result excludes the concentration of the C9-C10 aromatic range.
Avg	Results reported as a mathematical average.

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0812357

ESS LABORATORY CERTIFICATIONS

U.S. Army Corps of Engineers
Soil and Water

Rhode Island: A-179
Potable and Non Potable Water
<http://www.health.ri.gov/labs/waterlabs-instate.php>

Connecticut: PH-0750
Potable and Non Potable Water, Solid and Hazardous Waste
http://www.ct.gov/dph/lib/dph/environmental_health/environmental_laboratories/pdf/out_state.pdf

Maine: RI002
Potable and Non Potable Water
http://www.maine.gov/dep/blwq/topic/vessel/lab_list.pdf

Massachusetts: M-RI002
Potable and Non Potable Water
<http://public.dep.state.ma.us/labcert/labcert.aspx>

New Hampshire (NELAP accredited): 242405
Potable and Non Potable Water
<http://www4.egov.nh.gov/des/nhelap/namesearch.asp>

New York (NELAP accredited): 11313
Potable and Non Potable Water, Solid and Hazardous Waste
<http://www.wadsworth.org/labcert/elap/comm.html>

United States Department of Agriculture
Soil Permit: S-54210

New Jersey (NELAP accredited): RI002
Potable and Non Potable Water, Solid and Hazardous Waste
<http://www.nj.gov/dep/oqa/certlabs.htm>

Maryland: 301
Potable Water
http://www.mde.state.md.us/assets/document/wsp_labs

South Carolina: 78003
Volatile Organic Compounds in Potable Water

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CHAIN OF CUSTODY

Page _____ of _____

29

2018221

Turn Time ☒ Standard If faster than 5 days, prior approval by laboratory is required # State where samples were collected from: MA RI CT NH NJ NY ME Other Is this project for any of the following? MW-Water Navy USACE Other		Project # 212368 Project Name (to Client use) BPP Biogas BP Biogas ONE SERVICE RD		Zip 02905 PO# 143047 Email Address BPP Biogas @bpbio.com	
Co. Name MARRAGANSETH BAY COMMISSION BARRY WLENSKOWICZ City PROVIDENCE State RI		Telephone # (401) 461-8843 Fax # (401) 461-6540 Sample # 1 Date 11/10/07 Time 11:10 AM 2 Date 12/23/08 Time 2:10 PM 3 Date 12/23/08 Time 2:10 PM		Sample Identification (to Client use) 1 100 NBC BP BIOGAS FRONT 2 100 NBC BP BIOGAS BACK 3 100 NBC BP BIOGAS BACK	
ESS LAB Sample# 1 Date 12/23/08 Time 11:10 AM		CONF 1 LAB 1 MANT 1		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 2 Date 12/23/08 Time 11:10 AM		CONF 2 LAB 2 MANT 2		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 3 Date 12/23/08 Time 11:10 AM		CONF 3 LAB 3 MANT 3		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 4 Date 12/23/08 Time 11:10 AM		CONF 4 LAB 4 MANT 4		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 5 Date 12/23/08 Time 11:10 AM		CONF 5 LAB 5 MANT 5		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 6 Date 12/23/08 Time 11:10 AM		CONF 6 LAB 6 MANT 6		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 7 Date 12/23/08 Time 11:10 AM		CONF 7 LAB 7 MANT 7		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 8 Date 12/23/08 Time 11:10 AM		CONF 8 LAB 8 MANT 8		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 9 Date 12/23/08 Time 11:10 AM		CONF 9 LAB 9 MANT 9		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 10 Date 12/23/08 Time 11:10 AM		CONF 10 LAB 10 MANT 10		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 11 Date 12/23/08 Time 11:10 AM		CONF 11 LAB 11 MANT 11		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 12 Date 12/23/08 Time 11:10 AM		CONF 12 LAB 12 MANT 12		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 13 Date 12/23/08 Time 11:10 AM		CONF 13 LAB 13 MANT 13		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 14 Date 12/23/08 Time 11:10 AM		CONF 14 LAB 14 MANT 14		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 15 Date 12/23/08 Time 11:10 AM		CONF 15 LAB 15 MANT 15		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 16 Date 12/23/08 Time 11:10 AM		CONF 16 LAB 16 MANT 16		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 17 Date 12/23/08 Time 11:10 AM		CONF 17 LAB 17 MANT 17		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 18 Date 12/23/08 Time 11:10 AM		CONF 18 LAB 18 MANT 18		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 19 Date 12/23/08 Time 11:10 AM		CONF 19 LAB 19 MANT 19		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 20 Date 12/23/08 Time 11:10 AM		CONF 20 LAB 20 MANT 20		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 21 Date 12/23/08 Time 11:10 AM		CONF 21 LAB 21 MANT 21		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 22 Date 12/23/08 Time 11:10 AM		CONF 22 LAB 22 MANT 22		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 23 Date 12/23/08 Time 11:10 AM		CONF 23 LAB 23 MANT 23		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 24 Date 12/23/08 Time 11:10 AM		CONF 24 LAB 24 MANT 24		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 25 Date 12/23/08 Time 11:10 AM		CONF 25 LAB 25 MANT 25		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 26 Date 12/23/08 Time 11:10 AM		CONF 26 LAB 26 MANT 26		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 27 Date 12/23/08 Time 11:10 AM		CONF 27 LAB 27 MANT 27		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 28 Date 12/23/08 Time 11:10 AM		CONF 28 LAB 28 MANT 28		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 29 Date 12/23/08 Time 11:10 AM		CONF 29 LAB 29 MANT 29		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 30 Date 12/23/08 Time 11:10 AM		CONF 30 LAB 30 MANT 30		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 31 Date 12/23/08 Time 11:10 AM		CONF 31 LAB 31 MANT 31		Type of Containers 1 P Number of Containers 1 P	
ESS LAB Sample# 32 Date 12/23/08 Time 11:10 AM		CONF 32 LAB 3			

Please fax all changes to Chain of Custody in writing.

1 (White) Lab Copy 2 (Yellow) Client Receipt

ESS Laboratory Certificate of Analysis

Sample Date 1/29/2009

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

PROJECT NARRATIVE

Barry Wenskowicz
Narragansett Bay Commission
One Service Road
Providence, RI 02905

RE: BP Biogas
ESS Laboratory Work Order Number: 0901379

This signed Certificate of Analysis is our approved release of your analytical results. These results are only representative of sample aliquots received at the laboratory. ESS Laboratory expects its clients to follow all regulatory sampling guidelines. Beginning with this Project Narrative, the entire report has been paginated. The ESS Laboratory Certifications sheet is the final report page. This report should not be copied except in full without the approval of the laboratory. Samples will be disposed of thirty days after the final report has been delivered. If you have any questions or concerns, please feel free to call our Customer Service Department.

Laurel Stoddard
Laboratory Director

Digitally signed by Laurel Stoddard
Date: 2009.02.23 14:39:01 -05'00'

Analytical Summary

The project as described above has been analyzed in accordance with the ESS Quality Assurance Plan. This plan utilizes the following methodologies: US EPA SW-846, US EPA Methods for Chemical Analysis of Water and Wastes per 40 CFR Part 136, APHA Standard Methods for the Examination of Water and Wastewater, American Society for Testing and Materials (ASTM), and other recognized methodologies. The analyses with these noted observations are in conformance to the Quality Assurance Plan. In chromatographic analysis, manual integration may be used instead of automated integration because it produces more accurate results. All ICP Metals were analyzed using the established linear dynamic range to determine acceptable analytical results.

ESS Laboratory certifies that the test results meet the requirements of NELAC, except where noted within this project narrative.

Samples were analyzed in accordance with the Guidelines Establishing Test Procedures for the Analysis of Pollutants, 40 CFR Part 136, as amended.

Sample Receipt

The following sample(s) were received on January 29, 2009 for the analyses specified on the enclosed Chain of Custody Record.

<u>Laboratory ID</u>	<u>Matrix</u>	<u>Client Sample ID</u>
0901379-01	Waste Water	Front Biogas
0901379-02	Waste Water	Back Biogas

ESS Laboratory
Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0901379

PROJECT NARRATIVE

Classical Chemistry

BB90920-DUP1 Relative percent difference for duplicate is outside of criteria.
Silicon

No other observations noted.

End of Project Narrative.

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front Biogas
Date Sampled: 01/29/09 13:15
Percent Solids: N/A

ESS Laboratory Work Order: 0901379
ESS Laboratory Sample ID: 0901379-01
Sample Matrix: Waste Water

Classical Chemistry

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	2380	ug/sample	1010	3111D		10	JP	02/17/09

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CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front Biogas
Date Sampled: 01/29/09 13:15
Percent Solids: N/A
Initial Volume: 1
Final Volume: 20.25
Extraction Method: No Prep

ESS Laboratory Work Order: 0901379
ESS Laboratory Sample ID: 0901379-01
Sample Matrix: Waste Water
Analyst: VSC
Prepared: 02/04/09

Siloxane Analysis

Analyte	Results	Units	MRL	Method	Limit	DF	Analyst	Analzyed	I/V	F/V
Decamethylcyclopentasiloxane (D5)	3930	ug/Sample	1010	GC/MS		5	VSC	02/07/09	1	20.25
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	202	GC/MS		1	VSC	02/07/09	1	20.25
Hexamethyldisiloxane (MM)	ND	ug/Sample	202	GC/MS		1	VSC	02/07/09	1	20.25
Octamethylcyclotetrasiloxane (D4)	ND	ug/Sample	202	GC/MS		1	VSC	02/07/09	1	20.25

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Back Biogas
Date Sampled: 01/29/09 13:15
Percent Solids: N/A

ESS Laboratory Work Order: 0901379
ESS Laboratory Sample ID: 0901379-02
Sample Matrix: Waste Water

Classical Chemistry

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	135	ug/sample	94	3111D		1	JP	02/17/09

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Back Biogas
Date Sampled: 01/29/09 13:15
Percent Solids: N/A
Initial Volume: 1
Final Volume: 18.92
Extraction Method: No Prep

ESS Laboratory Work Order: 0901379
ESS Laboratory Sample ID: 0901379-02
Sample Matrix: Waste Water
Analyst: VSC
Prepared: 02/04/09

Siloxane Analysis

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>	<u>I/V</u>	<u>F/V</u>
Decamethylcyclopentasiloxane (D5)	ND	ug/Sample	189	GC/MS		1	VSC	02/07/09	1	18.92
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	189	GC/MS		1	VSC	02/07/09	1	18.92
Hexamethyldisiloxane (MM)	ND	ug/Sample	189	GC/MS		1	VSC	02/07/09	1	18.92
Octamethylcyclotetrasiloxane (D4)	ND	ug/Sample	189	GC/MS		1	VSC	02/07/09	1	18.92

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0901379

Quality Control Data

Analyte	Result	MRL	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Qualifier
Classical Chemistry										
Batch BB90920 - Metals No Prep										
Duplicate Source: 0901379-02										
Silicon	166	93.5	ug/sample	135				21	20	D+
Siloxane Analysis										
Batch BB90426 - No Prep										
Blank										
Decamethylcyclotrisiloxane (D5)	ND	200	ug/Sample							
Hexamethylcyclotrisiloxane (D3)	ND	200	ug/Sample							
Hexamethyldisiloxane (MM)	ND	200	ug/Sample							
Octamethylcyclotetrasiloxane (D4)	ND	200	ug/Sample							
Duplicate Source: 0901379-01										
Decamethylcyclotrisiloxane (D5)	4310	1010	ug/Sample	3930				9	20	
Hexamethylcyclotrisiloxane (D3)	ND	202	ug/Sample	ND					20	
Hexamethyldisiloxane (MM)	ND	202	ug/Sample	ND					20	
Octamethylcyclotetrasiloxane (D4)	108	202	ug/Sample	118				9	20	
Duplicate Source: 0901379-01										
Decamethylcyclotrisiloxane (D5)	4310	1010	ug/Sample	3930				9	20	
Hexamethylcyclotrisiloxane (D3)	ND	1010	ug/Sample	ND					20	
Hexamethyldisiloxane (MM)	ND	1010	ug/Sample	ND					20	
Octamethylcyclotetrasiloxane (D4)	ND	1010	ug/Sample	118					20

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0901379

Notes and Definitions

U	Analyte included in the analysis, but not detected
D+	Relative percent difference for duplicate is outside of criteria.
D	Diluted
ND	Analyte NOT DETECTED above the detection limit
dry	Sample results reported on a dry weight basis
RPD	Relative Percent Difference
MDL	Method Detection Limit
MRL	Method Reporting Limit
I/V	Initial Volume
F/V	Final Volume
§	Subcontracted analysis; see attached report
1	Range result excludes concentrations of surrogates and/or internal standards eluting in that range.
2	Range result excludes concentrations of target analytes eluting in that range.
3	Range result excludes the concentration of the C9-C10 aromatic range.
Avg	Results reported as a mathematical average.

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0901379

ESS LABORATORY CERTIFICATIONS

U.S. Army Corps of Engineers
Soil and Water

Rhode Island: A-179
Potable and Non Potable Water
<http://www.health.ri.gov/labs/waterlabs-instate.php>

Connecticut: PH-0750
Potable and Non Potable Water, Solid and Hazardous Waste
http://www.ct.gov/dph/lib/dph/environmental_health/environmental_laboratories/pdf/out_state.pdf

Maine: RI002
Potable and Non Potable Water
http://www.maine.gov/dep/blwq/topic/vessel/lab_list.pdf

Massachusetts: M-RI002
Potable and Non Potable Water
<http://public.dep.state.ma.us/labcert/labcert.aspx>

New Hampshire (NELAP accredited): 242405
Potable and Non Potable Water
<http://www4.egov.nh.gov/des/nhelap/namesearch.asp>

New York (NELAP accredited): 11313
Potable and Non Potable Water, Solid and Hazardous Waste
<http://www.wadsworth.org/labcert/elap/comm.html>

United States Department of Agriculture
Soil Permit: S-54210

New Jersey (NELAP accredited): RI002
Potable and Non Potable Water, Solid and Hazardous Waste
<http://www.nj.gov/dep/oqa/certlabs.htm>

Maryland: 301
Potable Water
http://www.mde.state.md.us/assets/document/wsp_labs

South Carolina: 78003
Volatile Organic Compounds in Potable Water

Page 1 of 1

Turn Time ☒ Standard If faster than 5 days, prior approval by laboratory is required. # _____ State where samples were collected from: MA RI CT NH NJ NY ME Other _____ I certify that this material for use of the following: _____	Reporting Limits Electronic Deliverable ☒ Yes ☐ No	ESS LAB PROJECT ID 0901379
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By circling MA-MCP, client acknowledges samples were collected
in accordance with MADEP CAM VII A

Please fax all changes to Chain of Custody in writing:

1 (White) Lab Copy 2 (Yellow) Client Receipt

10/29/2014 4

ESS Laboratory Certificate of Analysis

Sample Date 3/25/2009

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

PROJECT NARRATIVE

Barry Wenskowicz
Narragansett Bay Commission
One Service Road
Providence, RI 02905

RE: BP Biogas

ESS Laboratory Work Order Number: 0903287

This signed Certificate of Analysis is our approved release of your analytical results. These results are only representative of sample aliquots received at the laboratory. ESS Laboratory expects its clients to follow all regulatory sampling guidelines. Beginning with this Project Narrative, the entire report has been paginated. The ESS Laboratory Certifications sheet is the final report page. This report should not be copied except in full without the approval of the laboratory. Samples will be disposed of thirty days after the final report has been delivered. If you have any questions or concerns, please feel free to call our Customer Service Department.

Laurel Stoddard
Laboratory Director

Digitally signed by Laurel Stoddard
Date: 2009.04.02 15:54:06 -04'00'

Analytical Summary

The project as described above has been analyzed in accordance with the ESS Quality Assurance Plan. This plan utilizes the following methodologies: US EPA SW-846, US EPA Methods for Chemical Analysis of Water and Wastes per 40 CFR Part 136, APHA Standard Methods for the Examination of Water and Wastewater, American Society for Testing and Materials (ASTM), and other recognized methodologies. The analyses with these noted observations are in conformance to the Quality Assurance Plan. In chromatographic analysis, manual integration may be used instead of automated integration because it produces more accurate results. All ICP Metals were analyzed using the established linear dynamic range to determine acceptable analytical results.

ESS Laboratory certifies that the test results meet the requirements of NELAC, except where noted within this project narrative.

Samples were analyzed in accordance with the Guidelines Establishing Test Procedures for the Analysis of Pollutants, 40 CFR Part 136, as amended.

Sample Receipt

The following sample(s) were received on March 25, 2009 for the analyses specified on the enclosed Chain of Custody Record.

<u>Laboratory ID</u>	<u>Matrix</u>	<u>Client Sample ID</u>
0903287-01	Waste Water	Front
0903287-02	Waste Water	Back

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client-Project ID: BP Biogas

ESS Laboratory Work Order: 0903287

PROJECT NARRATIVE

No unusual observations noted.

End of Project Narrative.

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front
Date Sampled: 03/25/09 12:40
Percent Solids: N/A

ESS Laboratory Work Order: 0903287
ESS Laboratory Sample ID: 0903287-01
Sample Matrix: Waste Water

Classical Chemistry

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	RI - Permit <u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	1310	ug/sample	594	3111D		5	JP	03/30/09

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front
Date Sampled: 03/25/09 12:40
Percent Solids: N/A
Initial Volume: 1
Final Volume: 23.7
Extraction Method: No Prep

ESS Laboratory Work Order: 0903287
ESS Laboratory Sample ID: 0903287-01
Sample Matrix: Waste Water
Analyst: VSC
Prepared: 03/31/09

Siloxane Analysis

Analyte	Results	Units	MRL	Method	RI - Permit				
					Limit	DF	Analyst	Analyzed	I/V F/V
Decamethylcyclotrisiloxane (D5)	2280	ug/Sample	237	GC/MS		1	VSC	03/31/09	1 23.7
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	237	GC/MS		1	VSC	03/31/09	1 23.7
Hexamethyldisiloxane (MM)	ND	ug/Sample	237	GC/MS		1	VSC	03/31/09	1 23.7
Octamethylcyclotetrasiloxane (D4)	ND	ug/Sample	237	GC/MS		1	VSC	03/31/09	1 23.7

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Back
Date Sampled: 03/25/09 12:40
Percent Solids: N/A

ESS Laboratory Work Order: 0903287
ESS Laboratory Sample ID: 0903287-02
Sample Matrix: Waste Water

Classical Chemistry

Analyte
Silicon

Results
ND

Units
ug/sample

MRL
94

Method
3111D

RI - Permit

Limit

DF
1

Analyst
JP

Analyzed
03/30/09

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Back
Date Sampled: 03/25/09 12:40
Percent Solids: N/A
Initial Volume: 1
Final Volume: 18.7
Extraction Method: No Prep

ESS Laboratory Work Order: 0903287
ESS Laboratory Sample ID: 0903287-02
Sample Matrix: Waste Water
Analyst: VSC
Prepared: 03/31/09

Siloxane Analysis

<u>Analyte</u>	RI - Permit									
	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>	<u>L/V</u>	<u>F/V</u>
Decamethylcyclopentasiloxane (D5)	ND	ug/Sample	187	GC/MS		1	VSC	03/31/09	1	18.7
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	187	GC/MS		1	VSC	03/31/09	1	18.7
Hexamethyldisiloxane (MM)	ND	ug/Sample	187	GC/MS		1	VSC	03/31/09	1	18.7
Octamethylcyclotetrasiloxane (D4)	ND	ug/Sample	187	GC/MS		1	VSC	03/31/09	1	18.7

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0903287

Quality Control Data

Analyte	Result	MRL	Units	Spike Level	Source Result	%REC Limits	RPD Limit	Qualifier
Classical Chemistry								
Batch BC93013 - Metals No Prep								
Duplicate	Source: 0903287-02							
Silicon	ND	93.5	ug/sample	ND			20	
Siloxane Analysis								
Batch BC93123 - No Prep								
Blank								
Decamethylcyclopentasiloxane (D5)	ND	200	ug/Sample					
Hexamethylcyclotrisiloxane (D3)	ND	200	ug/Sample					
Hexamethyldisiloxane (MM)	ND	200	ug/Sample					
Octamethylcyclotetrasiloxane (D4)	ND	200	ug/Sample					
Duplicate	Source: 0903287-01							
Decamethylcyclopentasiloxane (D5)	2210	237	ug/Sample		2280		3	20
Hexamethylcyclotrisiloxane (D3)	ND	237	ug/Sample		ND			20
Hexamethyldisiloxane (MM)	ND	237	ug/Sample		ND			20
Octamethylcyclotetrasiloxane (D4)	146	237	ug/Sample		141		3	20

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0903287

Notes and Definitions

U	Analyte included in the analysis, but not detected
D	Diluted.
ND	Analyte NOT DETECTED above the detection limit
dry	Sample results reported on a dry weight basis
RPD	Relative Percent Difference
MDL	Method Detection Limit
MRL	Method Reporting Limit
I/V	Initial Volume
F/V	Final Volume
§	Subcontracted analysis; see attached report
1	Range result excludes concentrations of surrogates and/or internal standards eluting in that range.
2	Range result excludes concentrations of target analytes eluting in that range.
3	Range result excludes the concentration of the C9-C10 aromatic range.
Avg	Results reported as a mathematical average.

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0903287

ESS LABORATORY CERTIFICATIONS

U.S. Army Corps of Engineers
Soil and Water

Rhode Island: A-179
Potable and Non Potable Water
<http://www.health.ri.gov/labs/waterlabs-instate.php>

Connecticut: PH-0750
Potable and Non Potable Water, Solid and Hazardous Waste
http://www.ct.gov/dph/lib/dph/environmental_health/environmental_laboratories/pdf/out_state.pdf

Maine: RI002
Potable and Non Potable Water
http://www.maine.gov/dep/blwq/topic/vessel/lab_list.pdf

Massachusetts: M-RI002
Potable and Non Potable Water
<http://public.dep.state.ma.us/labcert/labcert.aspx>

New Hampshire (NELAP accredited): 242405
Potable and Non Potable Water
<http://www4.egov.nh.gov/des/nhelap/namesearch.asp>

New York (NELAP accredited): 11313
Potable and Non Potable Water, Solid and Hazardous Waste
<http://www.wadsworth.org/labcert/elap/comm.html>

United States Department of Agriculture
Soil Permit: S-54210

New Jersey (NELAP accredited): RI002
Potable and Non Potable Water, Solid and Hazardous Waste
<http://www.nj.gov/dep/oqa/certlabs.htm>

Maryland: 301
Potable Water
http://www.mde.state.md.us/assets/document/wsp_labs

South Carolina: 78003
Volatile Organic Compounds in Potable Water

ESS Laboratory

Division of Thielisch Engineering, Inc.
185 Frances Avenue, Cranston, RI 02910-2211
Tel. (401) 461-7181 Fax (401) 461-4486
www.esslaboratory.com

CHAIN OF CUSTODY

Page 1 of 1

ESS LAB PROJECT ID 78090		Reporting Limits Yes ___ No ___	
Electronic Deliverable Format: Excel ___ Access ___ PDF ___ Other ___		ESS LAB PROJECT ID 78090	
Turn Time If faster than 5 days, prior approval by laboratory is required # _____		ESS LAB PROJECT ID 78090	
State where samples were collected from: MA RI CT NH NJ NY ME Other _____		ESS LAB PROJECT ID 78090	
Is this project for any of the following: MA-MCP Navy USACE Other _____		ESS LAB PROJECT ID 78090	
Project # 02905		Project Name (20 Char or less) BP Bloggs	
Address One Service Rd		Address One Service Rd	
City Providence		City Providence	
State RI		State RI	
Zip 02905		Zip 02905	
Telephone # 401-461-8848		Telephone # 401-461-8848	
Fax # 401-461-6540		Fax # 401-461-6540	
Email Address Barry.Wenskowicz@NewBay.com		Email Address Barry.Wenskowicz@NewBay.com	
Sample Identification (20 Char or less) Front Back		Sample Identification (20 Char or less) Front Back	
ESS LAB Sample # 01 3/25/09 840-1240 ✓ 02 3/25/09 844-1240 ✓		ESS LAB Sample # 01 3/25/09 840-1240 ✓ 02 3/25/09 844-1240 ✓	
Date 3/25/09		Date 3/25/09	
Collection Time 840-1240		Collection Time 844-1240	
Container Type: P-Poly G-Glass S-Sterile V-VOA Matrx: S-Soil SD-Solid D-Sludge WW-Waste Water GW-Ground Water SW-Surface Water DW-Drinking Water O-Oil W-Wipes P-Filters		Container Type: P-Poly G-Glass S-Sterile V-VOA Matrx: S-Soil SD-Solid D-Sludge WW-Waste Water GW-Ground Water SW-Surface Water DW-Drinking Water O-Oil W-Wipes P-Filters	
Cooler Present ☒ Yes ___ No ___		Cooler Present ☒ Yes ___ No ___	
Seals Intact ☒ Yes ___ No ___		Seals Intact ☒ Yes ___ No ___	
Cooler Temp: 3.5		Cooler Temp: 3.5	
Relinquished by: (Signature) Barry Wenskowicz		Relinquished by: (Signature) Barry Wenskowicz	
Date/Time 3/25/09 1:20		Date/Time 3/25/09 1:20	
Received by: (Signature) Barry Wenskowicz		Received by: (Signature) Barry Wenskowicz	
Date/Time 3/25/09 1:20		Date/Time 3/25/09 1:20	
Relinquished by: (Signature) Barry Wenskowicz		Relinquished by: (Signature) Barry Wenskowicz	
Date/Time 3/25/09 1:20		Date/Time 3/25/09 1:20	

* By circling MA-MCP, client acknowledges samples were collected in accordance with MADEP CAM VII A

Please fax all changes to Chain of Custody in writing.

1 (White) Lab Copy 2 (Yellow) Client Receipt

10/28/04 B

ESS Laboratory Certificate of Analysis

Sample Date 5/29/2009

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0906005

PROJECT NARRATIVE

No unusual observations noted.

End of Project Narrative.

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front Biogas
Date Sampled: 05/29/09 13:20
Percent Solids: N/A

ESS Laboratory Work Order: 0906005
ESS Laboratory Sample ID: 0906005-01
Sample Matrix: Waste Water

Classical Chemistry

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	1150	ug/sample	215	3111D		2	JP	06/11/09

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front Biogas
Date Sampled: 05/29/09 13:20
Percent Solids: N/A
Initial Volume: 1
Final Volume: 21.5
Extraction Method: No Prep

ESS Laboratory Work Order: 0906005
ESS Laboratory Sample ID: 0906005-01
Sample Matrix: Waste Water
Analyst: VSC
Prepared: 06/05/09

Siloxane Analysis

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>	<u>I/V</u>	<u>F/V</u>
Decamethylcyclopentasiloxane (D5)	1520	ug/Sample	215	GC/MS		1	VSC	06/05/09	1	21.5
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	215	GC/MS		1	VSC	06/05/09	1	21.5
Hexamethyldisiloxane (MM)	ND	ug/Sample	215	GC/MS		1	VSC	06/05/09	1	21.5
Octamethylcyclotetrasiloxane (D4)	ND	ug/Sample	215	GC/MS		1	VSC	06/05/09	1	21.5

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Back Biogas
Date Sampled: 05/29/09 13:20
Percent Solids: N/A

ESS Laboratory Work Order: 0906005
ESS Laboratory Sample ID: 0906005-02
Sample Matrix: Waste Water

Classical Chemistry

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	ND	ug/sample	96	3111D		1	JP	06/11/09

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Back Biogas
Date Sampled: 05/29/09 13:20
Percent Solids: N/A
Initial Volume: 1
Final Volume: 21.5
Extraction Method: No Prep

ESS Laboratory Work Order: 0906005
ESS Laboratory Sample ID: 0906005-02
Sample Matrix: Waste Water
Analyst: VSC
Prepared: 06/05/09

Siloxane Analysis

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>	<u>I/V</u>	<u>F/V</u>
Decamethylcyclopentasiloxane (D5)	ND	ug/Sample	215	GC/MS		1	VSC	06/05/09	1	21.5
Hexamethylcyclotrisiloxane (D3)	ND	ug/Sample	215	GC/MS		1	VSC	06/05/09	1	21.5
Hexamethyldisiloxane (MM)	ND	ug/Sample	215	GC/MS		1	VSC	06/05/09	1	21.5
Octamethylcyclotetrasiloxane (D4)	ND	ug/Sample	215	GC/MS		1	VSC	06/05/09	1	21.5

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front Biogas - Blank
Date Sampled: 05/29/09 13:20
Percent Solids: N/A

ESS Laboratory Work Order: 0906005
ESS Laboratory Sample ID: 0906005-03
Sample Matrix: Waste Water

Classical Chemistry

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	ND	ug/sample	81	3111D		1	JP	06/11/09

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Back Biogas - Blank
Date Sampled: 05/29/09 13:20
Percent Solids: N/A

ESS Laboratory Work Order: 0906005
ESS Laboratory Sample ID: 0906005-04
Sample Matrix: Waste Water

Classical Chemistry

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRL</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	ND	ug/sample	93	3111D		1	JP	06/11/09

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas
Client Sample ID: Front Biogas - Spike
Date Sampled: 05/29/09 13:20
Percent Solids: N/A

ESS Laboratory Work Order: 0906005
ESS Laboratory Sample ID: 0906005-05
Sample Matrix: Waste Water

Classical Chemistry

<u>Analyte</u>	<u>Results</u>	<u>Units</u>	<u>MRI</u>	<u>Method</u>	<u>Limit</u>	<u>DF</u>	<u>Analyst</u>	<u>Analyzed</u>
Silicon	1360	ug/sample	198	3111D		2	JP	06/11/09

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0906005

Quality Control Data

Analyte	Result	MRL	Units	Spike Level	Source Result	%REC Limits	RPD	RPD Limit	Qualifier
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Classical Chemistry

Batch BF91101 - Metals No Prep

Duplicate Source: 0906005-05

Silicon	1290	198	ug/sample	1360			5	20	
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Siloxane Analysis

Batch BF90507 - No Prep

Blank

Decamethylcyclopentasiloxane (D5)	ND	10	ug/Sample
Hexamethylcyclotrisiloxane (D3)	ND	10	ug/Sample
Hexamethyldisiloxane (MM)	ND	10	ug/Sample
Octamethylcyclotetrasiloxane (D4)	ND	10	ug/Sample

Blank

Decamethylcyclopentasiloxane (D5)	ND	10	ug/Sample
Hexamethylcyclotrisiloxane (D3)	ND	10	ug/Sample
Hexamethyldisiloxane (MM)	ND	10	ug/Sample
Octamethylcyclotetrasiloxane (D4)	ND	10	ug/Sample

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0906005

Notes and Definitions

U	Analyte included in the analysis, but not detected
D	Diluted.
ND	Analyte NOT DETECTED above the detection limit
dry	Sample results reported on a dry weight basis
RPD	Relative Percent Difference
MDL	Method Detection Limit
MRL	Method Reporting Limit
I/V	Initial Volume
F/V	Final Volume
§	Subcontracted analysis; see attached report
1	Range result excludes concentrations of surrogates and/or internal standards eluting in that range.
2	Range result excludes concentrations of target analytes eluting in that range.
3	Range result excludes the concentration of the C9-C10 aromatic range.
Avg	Results reported as a mathematical average.

185 Frances Avenue, Cranston, RI 02910-2211

Tel: 401-461-7181

Fax: 401-461-4486

<http://www.ESSLaboratory.com>

Dependability

• Quality

• Service

11

ESS Laboratory

Division of Thielsch Engineering, Inc.

CERTIFICATE OF ANALYSIS

Client Name: Narragansett Bay Commission
Client Project ID: BP Biogas

ESS Laboratory Work Order: 0906005

ESS LABORATORY CERTIFICATIONS

U.S. Army Corps of Engineers
Soil and Water

Rhode Island: A-179
Potable and Non Potable Water
<http://www.health.ri.gov/labs/waterlabs-instate.php>

Connecticut: PH-0750
Potable and Non Potable Water, Solid and Hazardous Waste
http://www.ct.gov/dph/lib/dph/environmental_health/environmental_laboratories/pdf/out_state.pdf

Maine: RI002
Potable and Non Potable Water
http://www.maine.gov/dep/blwq/topic/vessel/lab_list.pdf

Massachusetts: M-RI002
Potable and Non Potable Water
<http://public.dep.state.ma.us/labcert/labcert.aspx>

New Hampshire (NELAP accredited): 242405
Potable and Non Potable Water
<http://www4.egov.nh.gov/des/nhelap/namesearch.asp>

New York (NELAP accredited): 11313
Potable and Non Potable Water, Solid and Hazardous Waste
<http://www.wadsworth.org/labcert/elap/comm.html>

United States Department of Agriculture
Soil Permit: S-54210

New Jersey (NELAP accredited): RI002
Potable and Non Potable Water, Solid and Hazardous Waste
<http://www.nj.gov/dep/oqa/certlabs.htm>

Maryland: 301
Potable Water
http://www.mde.state.md.us/assets/document/wsp_labs

South Carolina: 78003
Volatile Organic Compounds in Potable Water

ESS Laboratory
 Division of Thidisch Engineering, Inc.
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 Tel. (401) 461-7181 Fax (401) 461-4486
 www.esslaboratory.com

CHAIN OF CUSTODY

Page 1 of 1

Turn Time Standard Other ESS LAB PROJECT ID
 If faster than 5 days, prior approval by laboratory is required # 070605
 State where samples were collected from: MA RI CT NH NJ NY ME Other Other
 Is this project for any of the following: Navy USACE Other Other
 MA-MCP

Co. Name Narragansett Bay Commission Project # BP Biogeo Project Name (to Client or last) USACE Other

Contact Person Benny Wenskowicz Address One Service Rd Zip 02905 PO# PO#

City Providence State RI Email Address Benny.Wenskowicz@narrabay.com

Telephone # (401) 461-8848 x329 Fax # (401) 461-6540

ESS LAB Sample # 117 Date 5/29/09 Collection Time 10:20-12:00 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 118 Date 5/29/09 Collection Time 12:20-1:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 119 Date 5/29/09 Collection Time 1:50-3:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 120 Date 5/29/09 Collection Time 3:20-4:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 121 Date 5/29/09 Collection Time 4:50-6:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 122 Date 5/29/09 Collection Time 6:20-7:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 123 Date 5/29/09 Collection Time 7:50-9:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 124 Date 5/29/09 Collection Time 9:20-10:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 125 Date 5/29/09 Collection Time 10:50-12:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 126 Date 5/29/09 Collection Time 12:20-1:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 127 Date 5/29/09 Collection Time 1:50-3:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 128 Date 5/29/09 Collection Time 3:20-4:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 129 Date 5/29/09 Collection Time 4:50-6:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 130 Date 5/29/09 Collection Time 6:20-7:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 131 Date 5/29/09 Collection Time 7:50-9:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 132 Date 5/29/09 Collection Time 9:20-10:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 133 Date 5/29/09 Collection Time 10:50-12:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 134 Date 5/29/09 Collection Time 12:20-1:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 135 Date 5/29/09 Collection Time 1:50-3:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 136 Date 5/29/09 Collection Time 3:20-4:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 137 Date 5/29/09 Collection Time 4:50-6:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 138 Date 5/29/09 Collection Time 6:20-7:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 139 Date 5/29/09 Collection Time 7:50-9:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 140 Date 5/29/09 Collection Time 9:20-10:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

ESS LAB Sample # 141 Date 5/29/09 Collection Time 10:50-12:20 Matrix GRAB COMP ✓ Sample Identification (to Client or last) FRONT BIOGAS

ESS LAB Sample # 142 Date 5/29/09 Collection Time 12:20-1:50 Matrix GRAB COMP ✓ Sample Identification (to Client or last) BACK BIOGAS

Container Type: P-Poly G-Glass S-Sterile V-VOA Matrix: S-Soil D-Sludge W-Waste Water GW-Ground Water SW-Surface Water DW-Drinking Water O-Oil W-Wipes Y-Filters

Cooler Present ✓ Yes ✓ No ✓ Internal Use Only ✓ Yes ✓ No ✓ NA: ✓ [] Pickup

Seals Intact ✓ Yes ✓ No ✓ NA: ✓ [] Technicians ✓

Cooler Temp: 5/29/09 13:00 Relinquished by (Signature) Benny Wenskowicz Date/Time 5/29/09 15:00 Relinquished by: (Signature) Benny Wenskowicz Date/Time 5/29/09 15:00

Comments: Please report total mass - 0.21g sample F+8

Relinquished by (Signature) Benny Wenskowicz Date/Time 5/29/09 13:00 Relinquished by: (Signature) Benny Wenskowicz Date/Time 5/29/09 15:00

Relinquished by (Signature) Benny Wenskowicz Date/Time 5/29/09 13:00 Relinquished by: (Signature) Benny Wenskowicz Date/Time 5/29/09 15:00

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Relinquished by (Signature) Benny Wenskowicz Date/Time 5/29/09 13:00 Relinquished by: (Signature) Benny Wenskowicz Date/Time 5/29/09 15:00

*By checking MA-MCP, client acknowledges samples were collected in accordance with MADEP CAM VII A

Please fax all changes to Chain of Custody in writing.

1 (White) Lab Copy 2 (Yellow) Client Receipt

Rhode Island Analytical Certificate of Analysis

Sample Date 6/2/2009

CERTIFICATE OF ANALYSIS

Narragansett Bay Commission
Attn: Mr. Barry Wenskowicz
One Service Road
Providence, RI 02905

Date Received: 6/2/2009
Date Reported: 6/23/2009
P.O. #: 345180
Work Order #: 0906-09525

DESCRIPTION: BP BIOGAS

Subject sample(s) has/have been analyzed by our Warwick, R.I. laboratory and our subcontracted laboratory with the attached results.

Reference: All parameters analyzed by R.I. Analytical were by 40 CFR, 136 approved methodologies. The specific methodologies are listed in the methods column of the Certificate of Analysis.

Data qualifiers (if present) are explained in full at the end of a given sample's analytical results.

Certification #: RI-033, MA-RI015, CT-PH-0508, ME-RI015
NH-253700 A & B, USDA S-41844

If you have any questions regarding this work, or if we may be of further assistance, please contact us.

Approved by:

Data Reporting

enc: Chain of Custody

R.I. Analytical Laboratories, Inc.
 CERTIFICATE OF ANALYSIS

Narragansett Bay Commission
 Date Received: 6/2/2009
 Work Order #: 0906-09525

Approved by:
 Data Reporting

Sample # 001
 SAMPLE DESCRIPTION: BP BIOGAS
 SAMPLE TYPE: GRAB

SAMPLE DATE/TIME: 6/02/2009 @ 14:00.

PARAMETER	SAMPLE RESULTS	DET. LIMIT	UNITS	METHOD	DATE ANALYZED	ANALYST
BTU-HHV	See Attached			ASTM D5373	6/3/2009	SUB

LABORATORY REPORT

June 18, 2009

Data Reporting
RI Analytical Laboratories, Inc.
41 Illinois Avenue
Warwick, RI 02888

RE: 0906-09525

Dear Data Reporting:

Enclosed are the results of the sample submitted to our laboratory on June 3, 2009. For your reference, these analyses have been assigned our service request number P0901893.

All analyses were performed according to our laboratory's NELAP-approved quality assurance program. The test results meet requirements of the current NELAP standards, where applicable, and except as noted in the laboratory case narrative provided. For a specific list of NELAP-accredited analytes, refer to the certifications section at www.caslab.com. Results are intended to be considered in their entirety and apply only to the samples analyzed and reported herein. Your report contains 6 pages.

Columbia Analytical Services, Inc. is certified by the California Department of Health Services, NELAP Laboratory Certificate No. 02115CA; Arizona Department of Health Services, Certificate No. AZ0694; Florida Department of Health, NELAP Certification E871020; New Jersey Department of Environmental Protection, NELAP Laboratory Certification ID #CA009; New York State Department of Health, NELAP NY Lab ID No: 11221; Oregon Environmental Laboratory Accreditation Program, NELAP ID: CA20007; The American Industrial Hygiene Association, Laboratory #101661; Department of the Navy (NFESC); Pennsylvania Registration No. 68-03307; TX Commission of Environmental Quality, NELAP ID T104704413-08-TX. Each of the certifications listed above have an explicit Scope of Accreditation that applies to specific matrices/methods/analytes; therefore, please contact me for information corresponding to a particular certification.

If you have any questions, please call me at (805) 526-7161.

Respectfully submitted,

Columbia Analytical Services, Inc.

Kate Aguilera
Project Manager

Page
1 of 6

Client: RI Analytical Laboratories, Inc.
Project: 0906-09525

CAS Project No: P0901893

CASE NARRATIVE

The sample was received intact under chain of custody on June 3, 2009 and were stored in accordance with the analytical method requirements. Please refer to the sample acceptance check form for additional information. The results reported herein are applicable only to the condition of the sample at the time of sample receipt.

BTU and CHONS Analysis

The results for BTU and CHONS were generated according to ASTM D 3588-98. The following analyses were performed and used to calculate the BTU and CHONS results.

C2 through C6 Hydrocarbon Analysis

The sample was analyzed according to modified EPA Method TO-3 for C₂ through >C₆ hydrocarbons using a gas chromatograph equipped with a flame ionization detector (FID).

Fixed Gases Analysis

The sample was also analyzed for fixed gases (hydrogen, oxygen/argon, nitrogen, carbon monoxide, methane and carbon dioxide) according to modified EPA Method 3C (single injection) using a gas chromatograph equipped with a thermal conductivity detector (TCD).

Hydrogen Sulfide Analysis

The sample was also analyzed for hydrogen sulfide per modified SCAQMD Method 307-91 and ASTM D 5504-01 using a gas chromatograph equipped with a sulfur chemiluminescence detector (SCD).

The results of analyses are given in the attached laboratory report. All results are intended to be considered in their entirety, and Columbia Analytical Services, Inc. (CAS) is not responsible for utilization of less than the complete report.

Client: RI Analytical Laboratories, Inc.
Project: 0906-09525

Service Request: P0901893

SAMPLE CROSS-REFERENCE

<u>SAMPLE #</u>	<u>CLIENT SAMPLE ID</u>	<u>DATE</u>	<u>TIME</u>
P0901893-001	0906-09525-001 BP Biogas	6/2/09	14:00

Columbia Analytical Services, Inc.
Sample Acceptance Check Form

Client: RI Analytical Laboratories, Inc. Work order: P0901893
Project: 0906-09525
Sample(s) received on: 06/03/09 Date opened: 06/03/09 by: MZAMORA

Note: This form is used for all samples received by CAS. The use of this form for custody seals is strictly meant to indicate presence/absence and not as an indication of compliance or nonconformity. Thermal preservation and pH will only be evaluated either at the request of the client and/or as required by the method/SOP.

		Yes	No	N/A
1	Were sample containers properly marked with client sample ID?	☒	☐	☐
2	Container(s) supplied by CAS?	☒	☐	☐
3	Did sample containers arrive in good condition?	☒	☐	☐
4	Was a chain-of-custody provided?	☒	☐	☐
5	Was the chain-of-custody properly completed?	☒	☐	☐
6	Did sample container labels and/or tags agree with custody papers?	☒	☐	☐
7	Was sample volume received adequate for analysis?	☒	☐	☐
8	Are samples within specified holding times?	☒	☐	☐
9	Was proper temperature (thermal preservation) of cooler at receipt adhered to?	☐	☐	☒
	Cooler Temperature _____ °C Blank Temperature _____ °C			
10	Was a trip blank received?	☐	☒	☐
	Trip blank supplied by CAS: _____			
11	Were custody seals on outside of cooler/Box?	☐	☒	☐
	Location of seal(s)? _____ Sealing Lid?	☐	☐	☒
	Were signature and date included?	☐	☐	☒
	Were seals intact?	☐	☐	☒
	Were custody seals on outside of sample container?	☐	☒	☐
	Location of seal(s)? _____ Sealing Lid?	☐	☐	☒
	Were signature and date included?	☐	☐	☒
	Were seals intact?	☐	☐	☒
12	Do containers have appropriate preservation, according to method/SOP or Client specified information?	☐	☐	☒
	Is there a client indication that the submitted samples are pH preserved?	☐	☐	☒
	Were VOA vials checked for presence/absence of air bubbles?	☐	☐	☒
	Does the client/method/SOP require that the analyst check the sample pH and if necessary alter it?	☐	☐	☒
13	Tubes: Are the tubes capped and intact?	☐	☐	☒
	Do they contain moisture?	☐	☐	☒
14	Badges: Are the badges properly capped and intact?	☐	☐	☒
	Are dual bed badges separated and individually capped and intact?	☐	☐	☒

Lab Sample ID	Container Description	Required pH *	Received pH	Adjusted pH	VOA Headspace (Presence/Absence)	Receipt / Preservation Comments
P0901893-001.01	1.0 L Tedlar Bag					
P0901893-001.02	1.0 L Tedlar Bag					

Explain any discrepancies: (include lab sample ID numbers): _____

*Required pH: Phenols/COD/NH3/TOC/TOX/NO3+NO2/TKN/T.PHOS, H2SO4 (pH<2); Metals, HNO3 (pH<2); CN (NaOH or NaOH/Ace Acid) (pH>12); Diss. Sulfide, NaOH (pH>12); T. Sulfide, NaOH/Zn-Ac (pH>12)
P0901893 - RI Analytical Laboratories, Inc. 0906-09525 - Page 1 of 1

RSK - MEEP.P, HCL (pH<2); RSK - COL (pH 5-8); Sulfur (pH>4)

COLUMBIA ANALYTICAL SERVICES, INC.

RESULTS OF ANALYSIS

Page 1 of 1

Client: RI Analytical Laboratories, Inc.

Client Sample ID: 0906-09525-001 BP Biogas

Client Project ID: 0906-09525

CAS Project ID: P0901893

CAS Sample ID: P0901893-001

Test Code: ASTM D3588-98

Analyst: Zheng Wang/Wade Henton/Chris Cornett

Sampling Media: 1.0 L Tedlar Bag

Test Notes:

Date Collected: 6/2/09

Date Received: 6/3/09

Components	Result Volume %	Result Weight %	Data Qualifier
Hydrogen	< 0.01	< 0.01	
Oxygen + Argon	0.25	0.29	
Nitrogen	0.97	1.01	
Carbon Monoxide	< 0.01	< 0.01	
Methane	60.45	36.02	
Carbon Dioxide	38.32	62.65	
Hydrogen Sulfide	< 0.01	< 0.01	
Ethane	< 0.01	< 0.01	
Propane	< 0.01	< 0.01	
Butanes	< 0.01	< 0.01	
Pentanes	< 0.01	< 0.01	
Hexanes	< 0.01	< 0.01	
> Hexanes	< 0.01	< 0.01	
TOTALS	99.99	99.99	
Components	Mole %	Weight %	
Carbon	23.54	44.08	
Hydrogen	57.62	9.06	
Oxygen + Argon	18.38	45.85	
Nitrogen	0.46	1.01	
Sulfur	< 0.10	< 0.10	
Specific Gravity (Air = 1)		0.9293	
Specific Volume	ft ³ /lb	14.10	
Gross Heating Value (Dry Gas @ 60 F, 14.696 psia)	BTU/ft ³	612.6	
Net Heating Value (Dry Gas @ 60 F, 14.696 psia)	BTU/ft ³	551.6	
Gross Heating Value (Water Saturated at 0.25636 psia)	BTU/ft ³	600.1	
Net Heating Value (Water Saturated at 0.25636 psia)	BTU/ft ³	540.3	
Gross Heating Value (Dry Gas @ 60 F, 14.696 psia)	BTU/lb	8,637.4	
Net Heating Value (Dry Gas @ 60 F, 14.696 psia)	BTU/lb	7,777.2	
Compressibility Factor "Z" (60 F, 14.696 psia)		0.9969	

P0901893_ASTMD3588_0906150845_SS.xls - Sample

Verified By: Re- Date: 6/16/09

BTU.XLT - Page No.:

6

CHA N OF CUSTODY RECORD				Project Information	
R.L. Analytical Laboratories, Inc.				Client Information	
41 Illinois Avenue Warwick, RI 02888 Tel: 800-937-2580 Fax: 401-738-1970				Company Name: <u>Narragansett Bay Commission</u>	
131 Coolidge St, Bldg. 2 Hudson, MA 01749 Tel: 888-228-3334 Fax: 978-568-0078				Address: <u>One Service Rd</u>	
Date Collected: <u>6/2/09 2:00 PM</u>				City / State / Zip: <u>Providence, RI 02905</u>	
Field Sample Identification: <u>BP BIOGAS</u>				Telephone: <u>401-461-8848</u>	
Date Collected: <u>6/2/09 2:00 PM</u>				Contact Person: <u>BARRY WENSKOWSKI</u>	
Grab or Composite				Date: <u>6/2/09 2:50</u>	
# of Containers & Type				Time: <u>14:50</u>	
Preservation Code				Received By: <u>[Signature]</u>	
Matrix Code				Date: <u>6/2/09</u>	
03588-98				Time: <u>14:50</u>	
m.w. 1.57M				Project Name: <u>BP BIOGAS</u>	
BT				P.O. Number: <u>345180</u>	
x				Project Number: <u>461-6540</u>	
				Report To: <u>B. Wenskowski</u>	
				Sampled by: <u>B. Wenskowski</u>	
				Quote No: <u>NB 090546</u>	
				Email Address: <u>NarragansettBay.com</u>	
				Turn Around Time:	
				Normal ☒ EMAIL Report	
				5 Business days. Possible surcharge.	
				Rush ☐ (business days)	
				Lab Use Only	
				Sample Pick Up Only	
				RIAL sample; attach field hours	
				Shipped on ice <u>N/A</u>	
				Workorder No: <u>0905-09525</u>	
				M=MeOH, T=Na ₂ S ₂ O ₈ , Z=ZnOAc, I=Ice	
				Page of	

Project Comments			
Circle if applicable:	GW-1, GW-2, GW-3, S-1, S-2, S-3	MCP Data Enhancement	OG Package? Yes No
Container Types: P=Poly, G=Glass, AG=Amber Glass, V=Vial, St=Sterile Matrix Codes: GW=Groundwater, SW=Surface Water, WW=Wastewater, DW=Drinking Water, S=Soil, St=Sludge, A=Air, B=Bulk/Solid, O=			

Bibliography

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