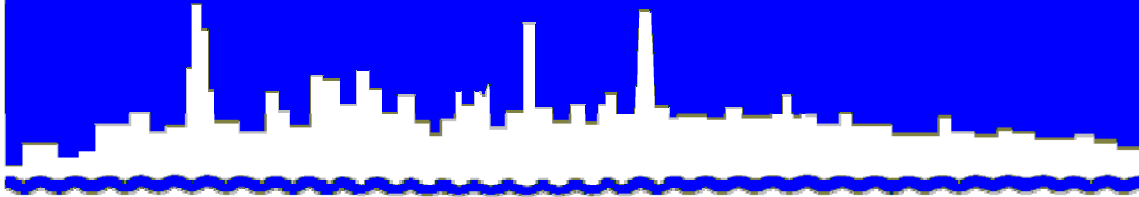


Protecting Our Water Environment



Metropolitan Water Reclamation District of Greater Chicago

**RESEARCH AND DEVELOPMENT
DEPARTMENT**

REPORT NO. 07-22

**SAMPLING AND ANALYSIS OF SILOXANE FROM THE DIGESTER
GAS LINE AT THE STICKNEY WATER RECLAMATION PLANT**

**PART OF MASTER PLAN PROJECT # 01-199-2P (INFRASTRUCTURE
AND PROCESS NEEDS FEASIBILITY STUDY)**

May 2007

Metropolitan Water Reclamation District of Greater Chicago

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DISCLAIMER

Mention of proprietary equipment and chemicals in this report does not constitute endorsement by the Metropolitan Water Reclamation District of Greater Chicago.

SUMMARY

The sampling and analysis of the digester gas for siloxane was conducted in response to Master Plan Project #01-199-2P (Infrastructure and Process Needs Feasibility Study) to evaluate the potential for deposit of siloxane in the boilers and turbine in the Stickney Water Reclamation Plant. The siloxane content of digester gas is a concern because it can form silicates during combustion. Silicates are very abrasive to equipment and reduce heat transfer in boilers. The results of this study will be used by the consultants to make decisions on whether a system to remove the siloxanes would be beneficial.

Sampling and analysis of siloxanes was carried out over the period of December 2005 through December 2006. The result of this study showed a wide variation in concentration of siloxanes in the digester gas line for the 14 sampling events during a one year period. Octamethylcyclotetrasiloxane (D4) and decamethylcyclopentasiloxane (D5) were the only siloxanes found in the digester gas. The concentrations varied from nondetectable to 12,000 $\mu\text{g}/\text{m}^3$ for D4 and nondetectable to 5,500 $\mu\text{g}/\text{m}^3$ for D5. These concentrations are approximately an order of magnitude lower than those detected by the Stickney Master Plan consultants.

Based on the study we have the following recommendations:

1. Further sampling should be conducted to verify differences in concentration observed by the District and Stickney Master Plan consultants.
2. Additional sampling should include simultaneous sampling at the compressor room and the boiler room so that variability in concentrations observed in this study at these points can be better understood.
3. Design for removal of siloxane from digester gas at the Stickney WRP should not be initiated until further sampling is completed.

INTRODUCTION

The Research and Development (R&D) Department has conducted a study to determine the concentration of siloxane in digester gas. The study included a series of sampling events of the digester gas line at three different locations in the Stickney Water Reclamation Plant (WRP), and contracted an outside laboratory to run the analysis for a series of siloxanes. The studies were requested by Stickney Master Plan Project Consultants (Black and Veatch [BV] and Greeley and Hansen [GH]) to evaluate the need for systems to remove the siloxanes from the digester gas line, because the Stickney Master Plan consultants detected siloxanes in their cursory investigation of the siloxane content of Stickney WRP digester gas.

The objective of this study was to determine the concentration of siloxane at various locations in the digester gas line, and to evaluate its seasonal variability.

This report presents information about the nature of siloxanes, method of sampling and analysis of siloxanes, and the results of analysis for siloxanes at the Stickney WRP.

Sampling for this study was conducted from December 2005 through December 2006. The sampling included 14 events and a total of 58 samples. Sampling of the digester gas was conducted once a month for 11 months except for three months in which the samples were collected twice.

The sampling and analysis protocol for this study was the same as that used by the consultants (BV and GH) in their initial study.

Siloxanes

Siloxanes are nontoxic organosilicones containing chains of silicon, oxygen and methyl groups. Siloxanes are manufactured in a wide variety of forms including low to high viscosity fluids, gums and resins. Siloxanes are found in commercial and consumer products such as detergents, shampoos, deodorants and cosmetics. Siloxanes can become incorporated in the digester gas at relatively high concentration during methane gas production. Siloxanes form silicon dioxide during digester gas combustion, which further combines with metals such as calcium, magnesium and aluminum in the gas stream to form silicates, the building blocks of glass and quartz-like material.

The emerging interest in siloxane is because the resultant silicates are very abrasive to moving parts and heated components in power generation equipment. Further, silicates reduce heat transfer capabilities in boilers, reduce flow through ported components, lower power production capacity, and can poison air emission catalysis.

A major problem with the use of digester gas for heat and power production has been silicate deposits. The primary cause of silicate-based material deposition is the increased use of siloxanes in consumer and industrial products over the past thirty years. During combustion of

digester gas containing siloxanes, silicon is released and combines with free oxygen or various other elements in the combustion gas. Deposits formed containing mostly silica and silicates (SiO_2 and SiO_3), but can also contain calcium, copper, sodium, sulfur and zinc. Most deposits caused by combustion of siloxanes are off-white to light brown in color and are of varying texture, some very smooth with a powdery-looking surface, while others are coarse and grainy. These deposits can ultimately build to a surface thickness of several millimeters, and are difficult to remove by chemical or mechanical means.

Siloxanes found in digester gas fall into two broad chemical compositions: linear and cyclical. The two most common linear siloxanes encountered are hexamethyldisiloxane (MM) and octamethyltrisiloxane (MDM). The two most common cyclical siloxanes found in digester gas are octamethylcyclotetrasiloxane (D4) and decamethylcyclopentasiloxane (D5). Another cyclic siloxane, dodecamethylcyclohexasiloxane (D6), was tested, but not found in the digester gas.

Method of Sampling for Siloxanes

Sampling for siloxanes was performed by dissolving the digester gas into two methanol filled impinger vials. The sampling train for siloxanes is depicted in Figure 1.

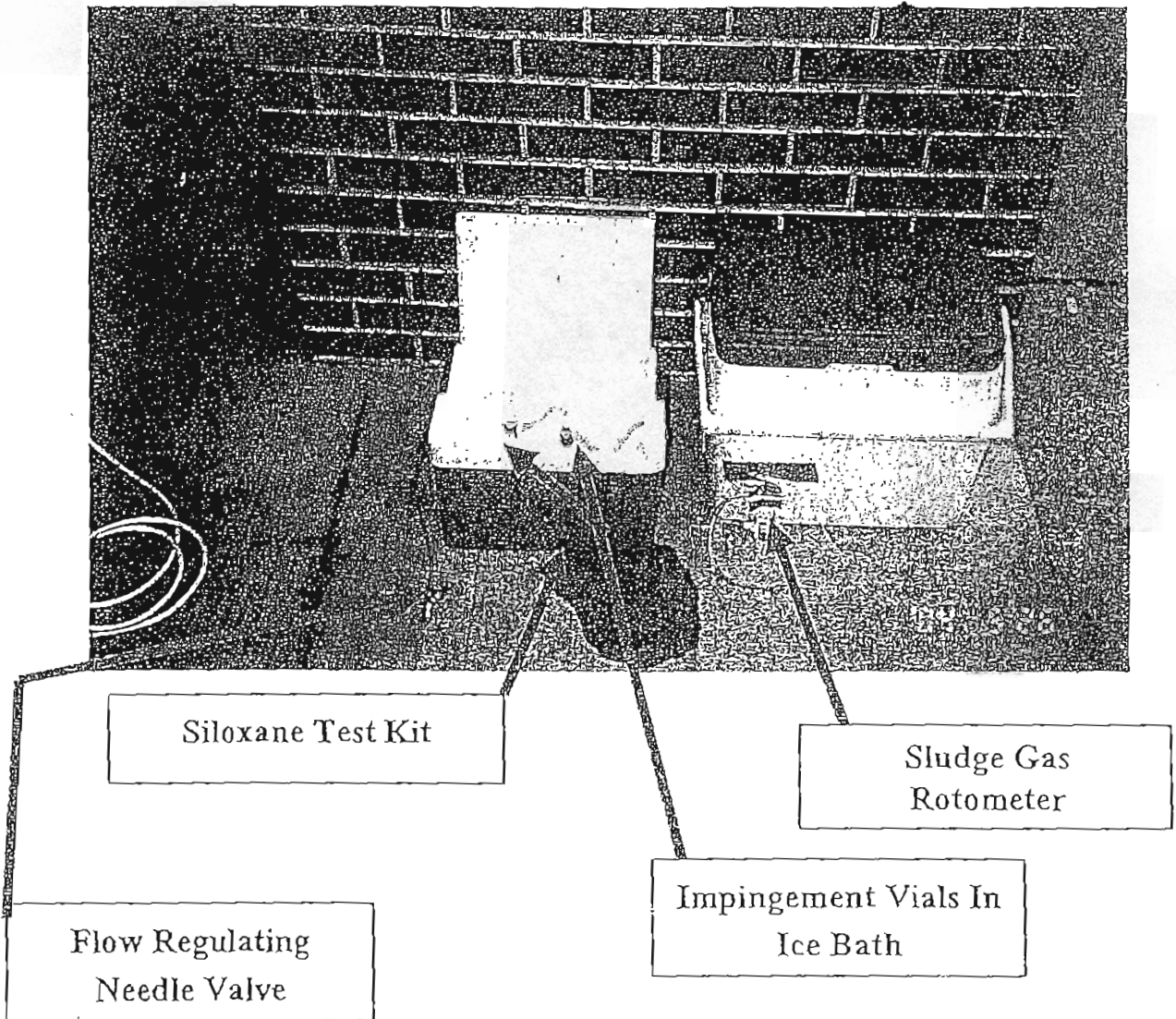
The sampling technique employed to collect siloxane included passing the digester gas at a prescribed flow rate (112 mL/min) through a pair of methanol filled vials connected to two midjet impingers (A and B) in accordance with the procedure provided by Air Toxics Ltd., Folsom, California. A schematic of the sampling train with sampling instructions is included in Appendix A. The impingers were immersed in an ice bath and the siloxane in the digester gas was absorbed in the chilled methanol. Each sample was collected over a 3-hr period. The samples were shipped overnight to Air Toxics Ltd for analysis of five siloxanes (D4, D5, D6, MM, and MDM). The sampling frequency was initially set at once per month for a 12-month period starting December 2005.

The sampling for siloxane was conducted in the same locations as was used by the consultants (BV and GH) in their initial study. The two sampling locations (Figures 2 and 3) which were selected by the consultants are as follows:

1. At the sludge gas compressor building on the compressor discharge header and upstream of the Sulfatreat units.
2. At the boiler building on the compressor discharge header and downstream of the Sulfatreat units.

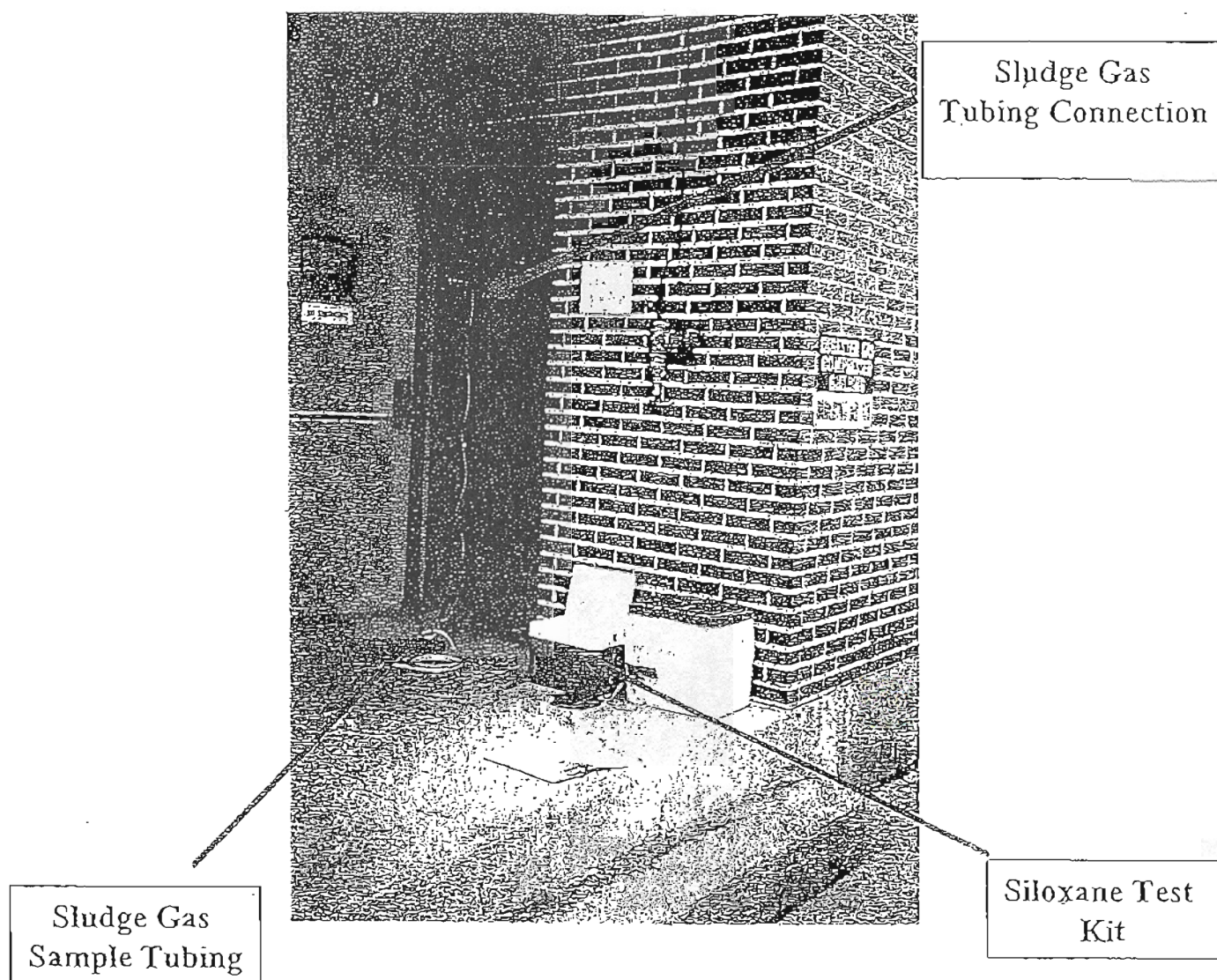
Upon initial evaluation of the results, a third location of the discharge at the outlet of the Sulfatreat unit was added in June 2006 in order to evaluate the possible effect of the Sulfatreat system on the observed increase in siloxane concentrations in the digester gas at the boiler room.

FIGURE 1: SILOXANE SAMPLING TRAIN*



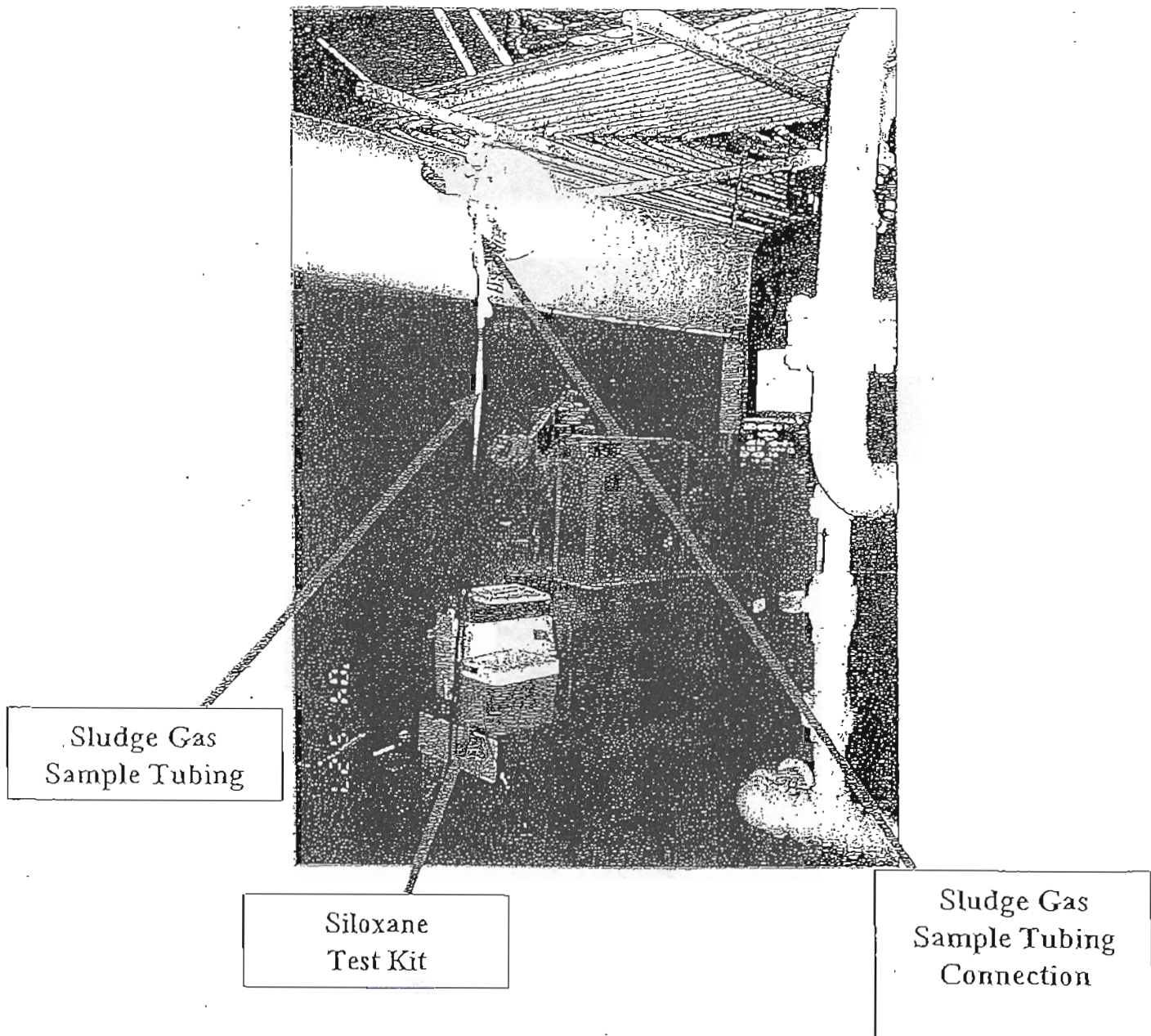
*Figure from report on Sludge Gas Siloxane Sampling & Analysis, August 2004 (BV and GH).

FIGURE 2: SAMPLING SILOXANE AT THE GAS COMPRESSOR BUILDING*



*Figure from report on Sludge Gas Siloxane Sampling & Analysis, August 2004 (BV and GH).

FIGURE 3: SAMPLING SILOXANE AT THE BOILER BUILDING*



*Figure from report on Sludge Gas Siloxane Sampling & Analysis,
August 2004 (BV and GH).

Sulfatreat System

The Sulfatreat unit consists of two chambers (South and North), and is designed to remove hydrogen sulfide from the gas stream. The compressed digester gas travels through both south and north vessels, in series, before traveling to the Central Heat (Boiler Room) for utilization in the boilers. The order of the path alternates, and is determined by the Sulfatreat media exchange schedule.

The H₂S concentration at the inlet and outlet of the Sulfatreat system was monitored by the Maintenance and Operations Department, and the results of this monitoring is shown in Table 1. The south and north Sulfatreat tanks were decommissioned and became nonoperational on 8/7/2006 and 9/28/2006, respectively.

Method of Siloxane Analysis

The sampling for siloxane followed the protocol provided by Air Toxics Ltd.

The analysis for siloxane was conducted by Air Toxics Ltd. (Folsom, California) using proprietary GC/MS method@71 (ATL@71). The GC/MS method builds upon the results of 1997 siloxanes investigation conducted by the Dow Corning Landfill Consortium. The Air Toxics method, in addition to using the Landfill Consortium method, has included specifications on sample collection, analysis and quality control, and has established a target compound list and the reporting limits.

The list of siloxanes analyzed by Air Toxics Inc. with the reporting limits is shown in Table 2.

TABLE 1: H₂S READINGS (ppm)

Date	Ambient Air Temperature °F	Compressor Room	Sulfatreat	
			North	South
1/19/06	45	550	0	95
2/14/06	42	700	0	300
3/3/06	37	500	0	220
3/17/06	36	400	0	150
4/5/06	60	300	0	145
7/13/06	76	210	0	120

8/7/06—South vessel decommissioned.

9/28/06—North vessel decommissioned.

TABLE 2: SILOXANE COMPOUNDS ANALYZED FOR
DIGESTER GAS AT STICKNEY WRP

Type of Siloxanes	Reporting Limit	
	Ppbv	$\mu\text{g}/\text{m}^3$
Octamethylcyclotetrasiloxane (D4)	130	1,100–1,500
Decamethylcyclopentasiloxane (D5)	100	1,100–1,600
Dodecamethylcyclohexasiloxane (D6)	180	2,200–3,200
Hexamethyldisiloxane (MM)	240	1,100–1,600
Octamethyltrisiloxane (MDM)	160	1,100–1,600

RESULTS OF ANALYSIS FOR SILOXANES

Fourteen sampling events were completed from December 2005 through December 2006, and the results of analysis are tabulated in Table 3. As shown in Table 3, only D4 and D5 were detected. The results show wide variations in concentration and on a number of occasions show an increase in concentration in the boiler room downstream of the Sulfatreat system samples as compared to the samples at the compressor room. Also, alternating the sampling time from AM to PM (Table 3) did not affect the general trend in siloxane concentrations between the compressor room and boiler room.

The range of measured concentrations for D4 was 1,500 and 3,500 $\mu\text{g}/\text{m}^3$ in the compressor room gas, and 1,600 and 7,700 $\mu\text{g}/\text{m}^3$ in the boiler room gas. D4 was not detected in 56% of the compressor room and boiler room gas samples.

D5 was observed in the boiler room gas and at the outlet of the Sulfatreat system but not in the compressor room gas, and the lowest and highest measured concentrations for boiler room gas were 1,300 and 5,500 $\mu\text{g}/\text{m}^3$, and 4,200 and 5,000 $\mu\text{g}/\text{m}^3$ at the outlet of the Sulfatreat system. The occurrence of nondetectable D5 in the digester gas at the boiler and at the outlet of the Sulfatreat system was 67%.

The sampling events of June to October 2006 included an additional sampling location immediately downstream of the Sulfatreat system. For the six sampling events at the outlet of the Sulfatreat system, the maximum observed concentrations of D4 and D5 were 12,000 and 5,000 $\mu\text{g}/\text{m}^3$, respectively.

Based on the results of the fourteen sampling events, a wide variation in concentration of siloxane across the Sulfatreat system and in the boiler room was observed. In one sampling event (7/25/2006), very high concentration of siloxane was found across the Sulfatreat system while the concentration at the compressor room ahead of Sulfatreat was below the reporting limit, and it implies that Sulfatreat could have contributed to formation of siloxane. In other instances, the concentration of siloxanes both at the compressor room and Sulfatreat outlet and the boiler room were all below the reporting limits, that in turn leaves a gap in understanding of the location of the formation of siloxanes in this system. As Table 3 shows, on 6/15/06 the concentrations of D4 at the compressor room, outlet of Sulfatreat system and boiler room were 3,500, 12,000 and 6,500 $\mu\text{g}/\text{m}^3$, respectively. This suggests that the concentration of D4 increased in the Sulfatreat system, and decreased while gas was traveling from Sulfatreat to the boiler room. However, this suggestion is speculative, because the sampling was not conducted simultaneously from all three locations. There is a possibility that the siloxane concentrations at one location varied during sampling of the other location. It was assumed that during the morning and afternoon hours of sampling the digester gas, the nature of the constituents and their concentrations had minimum fluctuations. However, the data indicate that this assumption was not correct.

The results of analysis for siloxanes in samples collected by the District generally were very low as compared to the results of analysis by the consultants (BV and GH). The consultants had reported the results of two sampling events that were carried out on 5/27/2004 and

TABLE 3: RESULTS OF SILOXANE ANALYSIS FOR STICKNEY WRP
DIGESTER GAS INCLUDING TIME OF SAMPLING

Date of Sample	Locations, Concentrations ($\mu\text{g}/\text{m}^3$), and Time of Sampling (AM/PM)								
	Compressor Room			Sulfatreat Outlet			Boiler Room		
	D4	D5	Time	D4	D5	Time	D4	D5	Time
12/7/2005	ND	ND	PM	—	—	—	ND	ND	AM
1/23/2006	1,600	ND	PM	—	—	—	4,000	1,400	AM
1/26/2006	ND	ND	PM	—	—	—	1,600	ND	AM
2/15/2006	2,300	ND	PM	—	—	—	4,200	1,300	AM
3/16/2006	1,500	ND	AM	—	—	—	7,700	5,500	PM
4/24/2006	ND	ND	PM	—	—	—	ND	ND	AM
5/25/2006	ND	ND	PM	—	—	—	ND	ND	AM
6/15/2006	3,500	ND	AM	12,000	4,200	PM	6,500	ND	PM
7/25/2006	ND	ND	AM	7,600	5,000	PM	—	—	—
7/26/2006	2,600	ND	AM	2,200	ND	PM	—	—	—
8/30/2006	ND	ND	PM	ND*	ND*	AM	—	—	—
10/3/2006	ND	ND	AM	ND**	ND**	PM	—	—	—
10/31/2006	ND	ND	AM	ND**	ND**	PM	—	—	—
12/19/2006	ND	ND	PM	—	—	—	ND**	ND**	AM

ND = Not detected.

— Not measured.

*South vessel not in service.

**South and north vessel not in service.

12/6/2004. The range of concentrations for D4 and D5 at the compressor room was 11,400 to 35,000 $\mu\text{g}/\text{m}^3$ and 18,800 to 76,000 $\mu\text{g}/\text{m}^3$, respectively. The range of concentrations for D4 and D5 at the boiler room was 8,930 to 31,300 $\mu\text{g}/\text{m}^3$ and 6,940 to 54,600 $\mu\text{g}/\text{m}^3$, respectively. The reason for the order of magnitude difference in the concentrations for siloxane reported by the consultants and the District is not known. However, the BV and GH analysis included only two days of analysis. On each of these days, D4 and D5 were detectable. In contrast, during the District's analysis, D4 and D5 were detected on three of 14 days sampled from the same locations that were sampled by the consultants (Table 4). This discrepancy must be addressed before siloxane removal is pursued.

Siloxane Removal Systems

Estimating the amount of silicates deposited on gas combustion systems due to siloxane is system specific. Determination of silicate deposits depends on the flow rate of digester gas into the combustion system, and the concentration of siloxanes. Based on a study by Applied Filter Technology (AFT, Snohomish, Washington), at least 0.1% to 0.5% of the combustion byproducts do not expel in the engine and turbine exhaust gas. Based on evaluation by AFT, if even a fraction of this small percentage deposit it might be sufficient to cause enough damage to an engine or turbine to require an overhaul.

Methods for removal of siloxanes from the digester gas are considered emerging technologies. Three different systems for siloxane removal from the digester gas stream were discussed in BV and GH's report titled "Sludge Gas Siloxane Sampling & Analysis, February 2005." The three systems are 1) high-flow SAGPack™, 2) hybrid high-flow SAGPack™ and SWOP™, and 3) low-flow SAGPack™, which are proprietary systems developed by AFT. These systems are designed to remove 95% to 100% of the siloxanes in the digester gas. BV and GH recommended the SWOP™ process in conjunction with the SAGPack™ system. The latter hybrid system was recommended to reduce the footprint of the removal system. The capital cost and M&O cost for these systems are shown in Table 5.

Other siloxane removal methods that have been recommended by the manufacturers of energy recovery systems such as Ingersoll-Rand and Capstone Turbine Corporation. These systems use a refrigerant gas drying system. In this method, the digester gas is chilled to below zero Fahrenheit, then passed through the carbon adsorption system. The refrigerant (chiller) usually removes the moisture up to 10%, and the overall siloxane removal could reach about 95%.

An estimate of capital and annual maintenance cost for siloxane removal system by Ingersoll-Rand (e-mail communications) is shown in Table 6. This estimate is based on gas flow rate in SCFM.

According to the District's operational data, the digester gas usage for year 2006 was about 2200 SCFM. The capital cost of installing a siloxane removal system for this flow rate is expected to fall below \$1000 per SCFM. A conservative estimate using \$1000 per cubic feet of digester gas will result in a capital cost of \$2,200,000, which is comparable to the capital cost

TABLE 4: COMPARISON BETWEEN THE RESULTS OF ANALYSIS FOR SILOXANES BY THE CONSULTANTS (BV AND GH) AND THE DISTRICT

Date of Sample	Locations and Concentrations ($\mu\text{g}/\text{m}^3$)									
	BV and GH					District*				
	Compressor Room		Boiler Room		Sulfatreat Outlet	Compressor Room		Boiler Room		Sulfatreat Outlet
D4	D5	D4	D5	D4		D5	D4	D5		
5/27/04	35,000	76,000	31,300	54,600	—	—	—	—	—	—
12/6/04	11,400	18,800	8,930	6,940	—	—	—	—	—	—
12/7/05	—	—	—	—	ND	ND	—	—	—	ND
1/23/06***	—	—	—	—	1,600	ND	—	—	—	4,000
1/26/06	—	—	—	—	ND	ND	—	—	—	1,600
2/15/06***	—	—	—	—	2,300	ND	—	—	—	4,200
3/16/06***	—	—	—	—	1,500	ND	—	—	—	7,700
4/24/06	—	—	—	—	ND	ND	—	—	—	ND
5/25/06	—	—	—	—	ND	ND	—	—	—	ND
6/15/06	—	—	—	—	3,500	ND	12,000	4,200	6,500	ND
7/25/06	—	—	—	—	ND	ND	7,600	5,000	—	—
7/26/06	—	—	—	—	2,600	ND	2,200	ND	—	—
8/30/06	—	—	—	—	ND	ND	ND*	ND*	—	—
10/3/06	—	—	—	—	ND	ND	ND**	ND**	—	—
10/31/06	—	—	—	—	ND	ND	ND**	ND**	—	—
12/19/06	—	—	—	—	ND	ND	—	—	ND**	ND**

ND = Not detected.

— = Not measured.

*South vessel not in service.

**North vessel not in service.

***D4 and D5 were not detected on 7 of the 14 days sampled by the District.

TABLE 5: CAPITAL COST AND ANNUAL SERVICE COST FOR AFT SILOXANE
REMOVAL SYSTEMS

Siloxane Removal System	Capital Cost	Annual Service Cost
SAGPack™ System-High Flow (5,760 scfm)	\$2,178,767	\$282,160
SAGPack™ and SWOP™-High Flow	\$1,230,636	\$73,900
SAGPack™ System-Low Flow (140 scfm)	\$138,528	\$17,940

TABLE 6: CAPITAL COST AND ANNUAL MAINTENANCE COST FOR INGERSOLL-RAND SILOXANE REMOVAL SYSTEM

Digester Gas Flow Rate (SCFM)	Capital Cost per SCFM	Annual Maintenance Cost
50-100	\$2,600	\$5,000
145-210	\$1,800	\$5,000
340-470	\$1,250	\$5,000

using AFT's high flow rate siloxane removal technology. The annual maintenance cost is not expected to be significantly higher.

Recommendations

Considering the major discrepancies in the concentration of siloxanes measured by the District and the consultants (BV and GH) at the compressor room and boiler room, it is recommended that the District, in coordination with a representative from the Master Planning Project Consultants (BV and GH), carry out simultaneous sampling at the compressor room and boiler room for a period of one month at a frequency of twice a week. Using this approach, it is expected to:

1. Detect the source of discrepancy between the measurement by the District and the consultants.
2. Develop a data set that will allow for more accurate comparison of the concentration of the siloxanes between the sampling locations.

APPENDIX A

AIR TOXICS LTD.
GUIDE FOR SILOXANES SAMPLING

@ 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 rotometer 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

AI-4

