THESIS

ANALYSIS OF OCTAMETHYLCYCLOTETRASILOXANE AND DECAMETHYLCYCLOPENTASILOXANE IN WASTEWATER, SLUDGE AND RIVER SAMPLES BY HEADSPACE GAS CHROMATOGRAPHY/MASS SPECTROMETRY

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ABSTRACT

ANALYSIS OF OCTAMETHYLCYCLOTETRASILOXANE AND DECAMETHYLCYCLOPENTASILOXANE IN WASTEWATER, SLUDGE AND RIVER SAMPLES BY HEADSPACE GAS CHROMATOGRAPHY/MASS SPECTROMETRY

Siloxanes are commonly used in cosmetic and personal care products, healthcare products and many industrial applications. Because siloxanes are persistent, they end up in the wastewater and go untreated through the wastewater treatment units, which lead to contamination of the surface waters through effluent discharge. Siloxanes tend to be adsorbed onto or absorbed by the activated sludge in the wastewater treatment process. In the digesters, the siloxanes volatilize and accumulate in the biogas, which leads to mechanical problems due to scaling. The two most common siloxanes detected in wastewaters and sludge are: octamethylcyclotetrasiloxane (D4) and decamethylcyclopentasiloxane (D5).

For this study, the D4 and D5 in wastewater and sludge samples were monitored using headspace gas chromatography/mass spectrometry. Samples were collected from the City of Loveland Wastewater Treatment Plant (WWTP), Loveland, CO and the Drake Wastewater Reclamation Facilities (WWRF), Fort Collins, CO. The levels of D4 were in the range of 0.7-11.3 $ng \cdot mL^{-1}$ in wastewater and 0.3-1.8 $\mu g \cdot g^{-1}$ dry solid in the sludge from Drake WWRF; 1.0-6.7 $ng \cdot mL^{-1}$ in wastewater and 0.3-1.7 $\mu g \cdot g^{-1}$ dry solid in the sludge from Loveland WWTP. D5 levels were determined in the range of 0.4-10.4 $ng \cdot mL^{-1}$ in wastewater and 3.2-31.4 $\mu g \cdot g^{-1}$ dry solid in the sludge from Drake WWRF; 0.5-14.0 $ng \cdot mL^{-1}$ in wastewater and 2.5-18.9 $\mu g \cdot g^{-1}$ dry solid in the sludge from Loveland WWTP. The concentrations of D4 and D5 were higher in this study compared to other researches in other countries and the concentrations in waste activated sludge

were in a comparable range. The concentrations of D4 and D5 in the receiving water body near the discharging points were below the limit of detection. The average mass loadings in the influent were 53.1 and 159.9 $g \cdot d^{-1}$ of D4 and 155.3 and 225.3 $g \cdot d^{-1}$ of D5 respectively in two plants.

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Chapter 1. Introduction

The siloxanes are widely used in the daily life. They are frequently added to consumer products like cosmetics, personal care products and healthcare products. They are also present in industrial applications (Dewil et al., 2006). Siloxanes in such products end up in the wastewater flowing into wastewater treatment plants (WWTPs). They have high volatility and low water solubility so they tend to accumulate in activated sludge. With temperature increase during anaerobic digestion, they volatilize and accrue in the biogas (Appels et al., 2008). They affect the performance of mechanical units by silica scaling, which will further increase the cost of biogas and energy generation.

With the increasing populations and the high demand for energy, the utilization of biogas is becoming one of the alternatives to the conventional fossil fuels. As a renewable energy, biogas can be beneficially used for the generation of electricity and heat. Biogas is formed by the anaerobic digestion of activated sludge in WWTPs and landfills. The anaerobic digestion also reduces the volume of activated sludge. The use of biogas for onsite generation of electricity can meet the WWTP's own demand, which significantly reduces the cost of operation. Besides, the use of biogas also decreases the potential of the global warming by preventing emission of biogas to atmosphere (Mcbean, 2008a). In the US, there were 1351 WWTPs which have flow rates lager than 1 MGD operating anaerobic digestion in 2011. However, only 8% of these plants generate biogas for beneficial use (US EPA, 2011). The presence of impurities in biogas reduces energy generation efficiency and causes some maintenance issues. One of the impurities are siloxanes, which represent a group of silicon containing organic compounds. During combustion of biogas, siloxanes convert to silica scaling and accumulate on the inner surfaces of equipment, which leads to combustion engine abrasion and damage and service interruptions. The presence of siloxanes increases the cost of operation and maintenance during the generation of energy by biogas.

Due to the occurrence of siloxanes in biogas, several removal methods have been developed, like adsorption and absorption (Schweighkofler et al, 2001). van Egmond et al. (2013) measured siloxanes in wastewater and sludge samples in a WWTP in UK. 11 WWTPs in Canada also reported the occurrence of siloxanes in influent, effluent and sludge samples (Wang et al., 2013). Removing siloxanes in the sludge and before they enter into the gaseous phase has not been fully studied. For the purpose of better evaluating the removal procedure, the analysis method of siloxanes in wastewater and sludge samples is being improved. A method for measuring siloxanes in surface water, wastewater and sludge samples was adopted and modified in this study. The concentrations of siloxanes from two utilities in Northern Colorado were also determined.

Chapter 2. Literature Review

2.1 Siloxanes

The name of siloxane comes from the nomenclature: Sil(icon) + ox(ygen) + (meth)ane (Dewil et al., 2006). Siloxanes represent a large group of silicones containing Si-O bonds with organic radicals bound to Si and including methyl, ethyl and other functional organic groups (UK Environment Agency, 2012). The structure of a siloxane can be linear and cyclic.

Today, siloxanes are widely used in various industrial processes, intermediate compounds for the production of other chemicals (silicone polymers), personal care products like fragrances, lotions, shampoos, deodorants, antiperspirants, skin cleansers, nail polishes, and consumer products such as detergents, paper coatings, and textiles (Wang et al, 2012; Dewil et al, 2006). . The annual worldwide production of siloxanes is estimated at over one million tons (Hagmann et al., 1999). About 54 tons of D4 and 1670 tons of D5 are discharged to wastewater, annually in Europe (Brooke et al., 2009). Table 2-1 indicates different uses of siloxanes (Dewil et al., 2006).

Siloxanes are commonly used because they are highly compressible, low flammable, have low surface tension, water repellent, high thermal stability. High temperature has limited effect on theirproperties. They have low toxicity and are not allergenic, which make them a favorable additive to the personal care products. Moreover, they are compliant with volatile organic compounds (VOC) restrictions. They are not environmentally persistent because they can be degraded in the atmosphere. They react with $OH^{\cdot}$ radicals most dominantly and NO_3 (nitrate ion) radicals and ozone (Wang et al., 2012). The calculated atmospheric half-lives of D4 and D5 were reported as ~10 d and ~20 d respectively, with the assumption that fist-order kinetics was applicable and that the tropospheric concentration of $OH^{\cdot}$ radicals was 7.7×105 molecule•cm^{-3}

over 24 h period (Atkinson, 1991). Table 2-2 illustrates the physical properties of the common siloxanes (Mcbean, 2008). The properties of methane are used as a comparison. Hexamethylcyclotrisiloxane (D3), Octamethylcyclotetrasiloxane (D4), Decamethylcyclopentasiloxane (D5) and Dodecamethylcyclohexasiloxane (D6) are cyclic volatile methylsiloxanes (cVMS), while Hexamethyldisiloxane (L2), Octamethyltrisiloxane (L3), Decamethyltetrasiloxane (L4) and Dodecamethylpentasiloxane (L5) are common linear siloxanes. cVMS have high vapor pressures, low water solubilities and high Henry's Law constants. Due to these properties, D4 is used as an off-site intermediate for the production of silicone polymers, at the same time D5 and D6 are mainly added to personal care products. Table 2-3 shows the structures of siloxanes (Kaj et al., 2004).

Table 2-1: Common Uses of Siloxanes and Silicones (Dewil et al., 2006)

Usage	Example
Medical usage	Implants in cosmetic surgery, tracheostomy tubes Coating hypodermic needles and bottle stops Coating pacemakers
Elastomer usage	Silicone components and tubing
Gels	Barrier creams Nappies
Adhesives	Fire retardants Extensive usage as carrier oils Waterproofing agents Fabric softeners Paints Penetrating oils Paper products Anti-foams Personal toiletries

Table 2-2: Physical Properties of Common Siloxanes (Mcbean, 2008)

Compound	Abbreviation	Molecular Weight ($g \cdot mol^{-1}$)	Boiling Point (℃)	Melting Point (℃)	Vapor Pressure (kPa @25℃)
Hexamethylcyclotrisiloxane C12H18O3Si3	D3	224.46	134	65	1.14
Octamethylcyclotetrasiloxane C8H24O4Si4	D4	296.61	175	17.4	0.13
Decamethylcyclopentasiloxane C10H3O5Si5	D5	370.77	210	-44	0.05
Dodecamethylcyclohexasiloxane C10H36O6Si6	D6	445	245	-3	0.003
Hexamethyldisiloxane C6H18Si2O	L2	162.4	100	-67	4.12
Octamethyltrisiloxane C8H24Si3O2	L3	236.5	153	-82	0.52
Decamethyltetrasiloxane C10H30Si4O3	L4	310.7	194	-68	0.073
Dodecamethylpentasiloxane C12H36Si5O4	L5	384.8	230	-81	0.009
Methane CH4	-	16.04	-162	-182	-

Table 2-3: Structures of Siloxanes (Kaj et al, 2004)

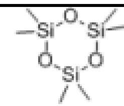
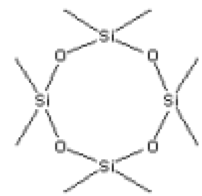
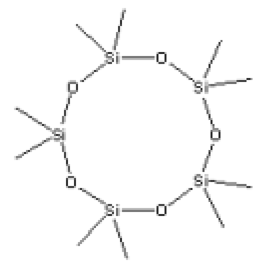
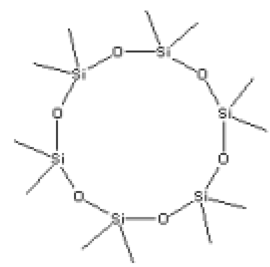
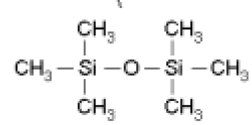
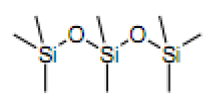
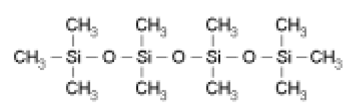
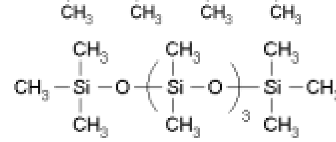
Abbreviation	CAS #	Structure
D3	541-05-9	
D4	556-67-2	
D5	541-02-6	
D6	540-97-6	
L2	107-46-0	
L3	107-51-7	
L4	141-64-8	
L5	141-63-9	

Table 2-4 shows the water solubilities of cyclic volatile methylsiloxanes in distilled water (Varaprath et al., 1996).

Table 2-4: Water Solubilities of Cyclic Volatile Methylsiloxanes(Varaprath et al., 1996)

Compound	Solubility ($\mu g \cdot L^{-1}$)
D4	56
D5	17
D6	5

The organic carbon partitioning coefficient (K_{OC}) is a physical parameter for describing transfer of chemicals between the water and sediment or water and soil exchange. K_{OC} is the ratio between the mass of a chemical which is adsorbed in the soil or sediment per unit mass of organic carbon in the soil and the chemical concentration in solution at equilibrium. Miller, (2007) reported that the value of $logK_{OC}$ of D4 in soil was 4.22. The values of $logK_{OC}$ of D5 was indicated by van Egmond and Sanders (2010) to be 5.6-5.7 for the natural soil, 5.2-5.4 for the natural sediment and 5.4-5.5 for the artificial sediment.

The octanol/water partition coefficient (K_{OW}) is defined as the ratio between the concentration of chemical in a unit volume of n-octanol (a non-polar solvent) and the concentration in a unit volume of water (a polar solvent) after the octanol and water have reached equilibrium (Smith et al., 1988). The values of $logK_{ow}$ of D4 and D5 were measured by Bruggeman et al. (1984) as 4.45 and 5.20, respectively, by a high performance liquid chromatography retention time method. Using a slow-stirring method for multi-phase quilibrium in a closed system, the values of $logK_{ow}$ of D4 and D5 were determined as 6.49 and 8.03 by Kozerski) (2007) and7.0 and 8.07 for D4 and D5, respectively by a dual-syringe system method for multi-phase equilibrium in a closed system by Kozerski and Shawl (2007). Because both $logK_{OC}$ and $logK_{OW}$ forD4 and D5 are greater than 4, they have a strong tendency to bind to soil and sediment with high organic matter.

2.2 Siloxanes in the Environment

Siloxanes mostly volatilize into the atmosphere from personal care product and health care products, where they are decomposed into silanols and various carbonyl compounds (Muller et al., 1995). Some of them end up in the wastewater and hence at the wastewater treatment plants (WWTPs), where they cannot be degraded by the activated sludge treatment employed by the WWTPs. Siloxanes are not very soluble in water but they tend to adsorb onto organics. After the activated sludge process, siloxanes stay in the wasted sludge that may be sent to sludge digesters for biogas production or further treatment of the sludge before disposal (Hayes et al., 2003). Due to higher temperatures in the anaerobic sludge digester, most of siloxanes volatilize and end up in the biogas (Mcbean, 2008b).

Siloxanes are hard to remove from wastewater completely, so they could be discharged into the surface water bodies. It was reported by Mueller et al. (1995) that 0.06-0.41 $\mu g \cdot L^{-1}$ of D4 was detected in effluent of WWTPs in US. About <0.06-3.7 $\mu g \cdot L^{-1}$ of D4 and <0.04-3.8 $\mu g \cdot L^{-1}$ of D5 in wastewater were determined in Nordic environment (Kaj et al., 2005b). <0.01-0.029 $\mu g \cdot L^{-1}$ of D5 were detected in two rivers in Eastern England (Sparham et al., 2008). <0.009-0.023 $\mu g \cdot L^{-1}$ of D4 and <0.027-1.48 $\mu g \cdot L^{-1}$ of D5 in a river in Canada (Wang et al., 2012). In the aquatic environment, siloxanes tend to be degraded very slowly and bioaccumulate in fish (Brooke et al., 2009). D5 was detected in fish in several surface waters which receive effluents from WWTPs in Nordic countries, such as the Oslo Fjord (Kaj et al, 2005a), the Rhine River (Brooke et al., 2009), and the Svalbard (Warner et al, 2010). Traveling with the surface waters, siloxanes were found in the flesh of the fish in other water bodies far from the effluent discharge points. For example, D4, D5 and D6 at 10, 200 and 40 $ng \cdot g^{-1}$ lipid weight concentration, respectively, were determined in herring in many locations in the Baltic Sea which didn't receive any effluent from

WWTP directly (Kierkegaard et al, 2012). As shown in Table 2-5, siloxanes were found in different species from lakes in Norway (Borga et al., 2013). D5 dominated the major contaminant among siloxanes in these species due to the large usage of D5. It was also reported that 0.8-14.4 ng·g^{-1} wet weight of D5 was present in perch from six Swedish lakes that received WWTP effluent (Kierkegaard et al., 2013). Bioaccumulation of siloxanes could occur to human by consumption of fish contaminated by siloxanes.

Table 2-5: Concentrations of Silxoanes in Some Species in Norwegian Lakes (Borga et al., 2013)

		Mean Concentrations (ng/g lipid weight)		
Lake	Spices	D4	D5	D6
Mjøsa	mysis	53	930	59
	vendace	81	14160	790
	smelt. small	24	3530	180
	smelt. large	17	5260	330
	brown trout	27	5630	290
Randsfjorden	whitefish	<19	110	<30
	smelt	<11	970	58
	brown trout	16	2700	140
Femunden	Arctic char	<10	<20	<40
	brown trout	<40	39	<80

Siloxanes are also present in atmosphere due to the usage and emission of biogas. In a study of biogas, 4800-5100 µg ·m^{-3} of D4 and 600-650 µg ·m^{-3} of D5 were determined in landfill gas, and 6300-8200 µg ·m^{-3} of D4 and 9400-15000 µg ·m^{-3} of D5 were reported in wastewater gas (Schweighkofler et al, 2001). D6 was reported with a concentration of 210 µg ·m^{-3} in the landfill gas in Canada (McBean, 2008b). Rasi et al., (2010) demonstrated that the amount of cyclic siloxanes in different places varied greatly because of the different raw waste and wastewater, different treatment processes together with the different biogas generation processes. Compared with biogas, the concentrations of siloxanes in indoor and outdoor air were much lesser. The common usage of cosmetic and personal care products dominated the typical source of siloxanes in indoor air (Gouin et al, 2013, Montemayor et al., 2013). Mean concentrations of D5 of 39.6 μg $\cdot m^{-3}$, 26.1 μg $\cdot m^{-3}$ and 7.0 μg $\cdot m^{-3}$ were detected in indoor air from administrative offices, data centers and telecommunication offices in US (Shields et al., 1996). D4, D5 and D6 were identified in 73, 250 and 142 out of 400 studied Swedish homes with mean concentrations of 9.0, 9.7 and 7.9 μg $\cdot m^{-3}$ (Kaj et al, 2005b). Another study conducted by Steer et al. (2008) in Canada reported that 0.1-0.2 μg $\cdot m^{-3}$ D4 were determined in non-lab and analytical lab air and about 0.03 μg $\cdot m^{-3}$ in clean labs. D5 concentrations were determined at 14 μg $\cdot m^{-3}$ in analytical laboratory and 9 μg $\cdot m^{-3}$ in non-lab locations. It was concluded that higher siloxanes concentrations were present in the locations close to WWTP and in enclosed buildings (Steer et al, 2008). Because of the diffusion, the concentrations of siloxanes in outdoor air are lower than that in indoor air. It was reported that D4 was determined at trace amounts in the US atmosphere in the mid-1970s (Pellizzari et al, 1976). D5 was detected in trace concentration in the air samples from China. D3 and D4 were measured with highest concentrations in an industrial area compared to the air samples from urban, industrial, landfill, WWTP, suburban and a forest park. All of these areas except the forest park were detected the occurrence of siloxanes (Wang et al, 2001). Yucuis et al. (2013) measured the outdoor air concentrations of siloxanes in areas in Chicago, US. It was reported that 54, 17 and 9.7 ng$\cdot m^{-3}$ of D4 and 210, 52, 18 ng$\cdot m^{-3}$ of D5 were detected in urban, suburban and rural area respectively.

2.3 Impacts of Siloxanes of Environment

The remaining siloxanes in the wastewater effluents may be discharged to surface waters. In water bodies, siloxanes exhibit little toxicity to the organisms because of their low water solubilities (Wang et al., 2013). The large molecular size of siloxanes reduces uptake by organism, but over time they will bioaccumulate. The equilibrium partitioning between the environment and organisms can be represented directly by the bioconcentration factor (BCF). The BCFs of 1090, 1010 and 1200 L·kg $^{-1}$ for D4, D5 and D6 respectively were determined for a goldfish exposure (Opperhuizen et al., 1987). Then the BCFs of 1875-10000 L·kg $^{-1}$ (Annelin and Frye, 1989) and 12400 L·kg $^{-1}$ (Fackler et al., 1995) for D4, 4450 L·kg $^{-1}$ (Parrott et al., 2013) and 13300 L·kg $^{-1}$ (Drottar, 2005a) for D5 were detected in fathead minnows studies. Drottar, 2005b reported that the BCF of 1660 L·kg $^{-1}$ for D6 was estimated using fathead minnow. These researches reported BCF values for D4 and D5 were larger than 2000, which indicated D4 and D5 were bioaccumulative (BCF >2000) (European Commission, 2010).

As a result of siloxanes' bioaccumulative property, they present toxicity to aquatic animals. Wang et al. (2012) studied the toxicity of D4 and demonstrated that D4 can be toxic to some aquatic creatures at very low concentrations (15 μg·L^{-1}) while other organisms were not affected. Sousa et A-(1995) showed that rainbow trout was the most sensitive species to D4 at10 μg·L^{-1}. Giesy et al. (2011) reported that D5 was not toxic to fish and water flea exposed at concentration up to its water solubility limit (17 μg·L^{-1}). Drottar et al. (2005) reported that D6 exhibited no biological adverse effects on fathead minnow under 49-day flow-through test conditions. The concentration of D6 analyzed was up to 4.6 μg·L^{-1}, which was close to its water solubility limit (5 μg·L^{-1}).

In sediments and soil, siloxanes present toxicity to organisms. A study of prolonged sediment toxicity on midges reported that the detected no-observed-effect-concentration (NOEC) for both percent survival and emergence were 44 μg·g^{-1}(Krueger et al, 2008a). . Sediments were spiked with D4 at concentrations from 6.5 to 355 μg·g^{-1}, and the midges exposed to the maximum concentration presented a statistically significant reduction in development. Another study about exposing midges to 160 μg·g^{-1} indicated that D5 presented a statistically significant reduction in development. The NOEC of D5 for midge development was 70 μg·g^{-1}(Krueger et al, 2008b).

Because siloxanes are detected at considerable levels in water, they are under consideration by UK Environment Agency and Canadian Environmental Assessment Agency for drinking water regulations and defined harmful to the environment by Environment Canada and Health Canada. The European Commission classified D4 as Category 3 for reproductive toxicity. It was determined by Dow Corning Corporation that D5 showed a potential carcinogenic effect in a 2-year chronic toxicity and carcinogenicity study (US EPA, 2009). In this 2-year research, 344 rats of 60 male and 60 female were exposed to vapor concentrations up to 450 mg·g^{-3} of D5 for 6 h per day, 5 d per week. The female rats exposed to 450 mg·g^{-3} of D5 presented a statistically significant increase of uterine tumors after 2-year exposure (US EPA, 2009).

There were not effects on human health by D4 in low concentrations reported. Looney et al, (1998) found there were no immunological effects on human volunteers by D4. In this study, 12 normal volunteers (8 males and 4 females) were exposed to 122 μg·L^{-1} D4 for 1 h and their blood samples were obtained for before, immediately after, and 1, 6, 24 h postexposure. This study concluded that immunotoxic or inflammatory effects of respiratory exposure to D4 were not found (Looney et al., 1998).

2.4 Impacts of Siloxanes on Wastewater Treatment Utilities

At the wastewater treatment utilities siloxanes affect the performance of various mechanical units due to scaling. During the combustion of the biogas, in the boilers, the siloxanes are converted into abrasive microcrystalline silica, which has chemical and physical properties similar to those of glass. This material deposits and accumulates on the surface of the mechanical units and becomes thicker. First, the hardness of this residue leads to the abrasion of gas motor surfaces. Then since siloxanes are thermal and electrical insulators, they decrease the heat transferring efficiency of boilers and fire tubes. Due to this layer, the demand of biogas increases to meet the same energy generation. Accumulation leads to the overheating of sensitive motor parts and depresses the function of spark plugs. It leads to serious motor damage and shortening of operation time (Appels et al., 2008). . Finally, frequent-monitoring, cleaning and repair of engines is required to prevent damages to valves, pistons rings, liners, cylinder head, spark plugs and turbocharges. Many wastewater treatment plants which utilize the biogas for energy production face these problems. In Trecatti (UK), a major engine failure was caused by the presence of less than 400 mg/m^3 of volatile siloxanes during 200 hours of operation(Griffin, 2004). Figure 2-1 illustrates the deposition of silica scaling in a boiler (Dewil et al., 2006).

Figure 2-1: Boiler Clogging by Silica Scaling (Dewil et al., 2006)

In US, 43% of wastewater treatment plants greater than 1 MGD (million gallons per day) apply anaerobic digestion but only 8% of them use biogas to generate electrical or thermal energy

(Water Environment Research Fundation, 2012). Increasing the efficiency together with the potential to generate renewable energy from wastewater is considerably significant. As a result, siloxanes are becoming more of a concern. The removal of siloxanes in the biogas and the activated sludge is needed.

2.5 Treatment/Romoval of Siloxanes

In US, 43% of wastewater treatment plants with greater than 1 MGD (million gallons per day) capacity apply anaerobic digestion but only 8% of them use biogas to generate electrical or thermal energy (WERF, 2012). Increasing the efficiency together with the potential to generate renewable energy from wastewater is considerably significant. As a result, siloxanes are becoming more of a concern. The removal of siloxanes in the biogas and the activated sludge is needed.

D4 and D5 occupy the major portion of the siloxanes in the biogas produced during anaerobic digestion of the sludge. L2 and L3 are not likely in the biogas because they are much easier to dissolve in the water compared to D4 and D5 (Zhang et al., 2007). There are several methods to remove siloxanes from the biogas including physical, chemical and biological methods.

Adsorption using activated carbon is able to reduce the siloxanes to reach the concentration of total silicone less than 0.1 $mg \cdot m^{-3}$ (Rossol et al., 2003). Other absorbents like alumina and silicone are also effective in removing siloxanes. Alumina has the adsorption capacity of 1.3 wt% to D4 (Lee et al., 2001).

Selexol™ (Poly(ethylene glycol) dimethyl ether) is a potential solvent, which can remove 99% of siloxanes in biogas (Wheless and Pierce, 2004). It is reported that sulfuric acid (concentration

≥ 48%) and nitric acid (concentration ≥65%) have removal efficiency of more than 95% for D5 (Schweighkofler et al, 2001).

Cryocondensation uses very low temperature to condense volatile siloxanes in the biogas. From the melting point shown in Table 2, it is easy to understand that with low enough temperature, most siloxanes transform to solid phase while the methane is still in gas phase. It was reported that under -70℃, the removal efficiency could reach 99.3% (Hagmann et al, 2001). However, to decrease the temperature needs a lot of energy, so it is feasible only for the biogas with a high concentration of siloxanes (Xu et al, 2012).

Biological degradation is also feasible for the removal siloxanes. But the degradation process takes months to reach low removal efficiency (Xu et al., 2012, Popat et al., 2008, Accettola et al., 2008) .

Peroxidation is a useful method for the removal of siloxanes from waste activated sludge (Appels et al., 2008). Three oxidants were analyzed and compared: hydrogen peroxide (H_2O_2), POMS (H_2SO_5) and dimethyldioxiranes (DMDO). All of methodologies showed an approximately 50% removal efficiency of D4 and D5, except for the DMDO peroxidation of D4, which can remove up to 85%.

2.6 Detection and Analysis of Siloxanes

Siloxanes have been measured in many matrices, like sludge, water and soil. Dewil et al. (2007) quantified D4 and D5 in sludge samples from the secondary clarifier. The siloxanes in the sludge were extracted with n-hexane. Then the siloxanes were analyzed by a Varian 3400 gas chromatograph coupled with a FID detector. A Varian Factor Four VF-1MS capillary column was used for the separation. The injector port temperature was set at 125 °C. The initial oven temperature was 60 °C which was held for 4 min. After that the temperature was linearly increased to 250 °C at a rate of 8 $°C·min^{-1}$. This temperature was kept for another 15 min. The detector temperature was set at 250 °C. The detected concentrations of D4 and D5 were 0.9 $mg·g^{-1}$ dry solid and 0.58 $mg·g^{-1}$ dry solid respectively (Dewil et al., 2007).

The GC/MS analysis was performed for the analysis of cyclic and linear siloxanes in soil by Sanchez-Brunete et al., (2010). In this study, siloxanes were extracted with n-hexane and quantified with a GC/MS equipped with a fused silica capillary column ZB-5MS. The injector port temperature was at 200 °C. Helium gas is the carrier gas which has a flow rate of 1.0 $mL·min^{-1}$. The initial oven temperature was 40 °C for 2 min. Then the temperature was increased to 220 °C. The mass spectrometric detector was used in electron impact ionization mode with an ion source temperature of 300 °C and a quadrupole temperature of 150 °C. The tetrakis(trimethylsilyloxy)silane (M4Q) was used as the internal standard. This study concluded that 9.2-56.9 $ng·g^{-1}$ of D5 and 5.8-27.1 $ng·g^{-1}$ of D6 were detected in agricultural soils and 22-184 $ng·g^{-1}$ of D5 and 28-483 $ng·g^{-1}$ of D6 in industrial soils. D4 were found in only one of those industrial soil sample with a concentration of 58.6 $ng·g^{-1}$, but not in any of the agricultural ones (Sanchez-Brunete et al., 2010).

Another analysis method was applied to determine the concentrations in soil and sludge samples which was concurrent solvent recondensation – large volume injection – GC/MS (CSR-LVI-GC/MS) done by Companioni-Damas et al. (2012). The extraction procedure was applied as following: First, 0.5 g sample equilibration stayed at 4 °C overnight. Second, it was mixed with 2 g of anhydrous sodium sulphate for 3 hours. Third, it was shook for 10 min with 3 mL n-hexane containing 0.2 g of activated Cu. Finally, it was centrifuged at 3500 rpm for 10 min. J&W DB-5MS capillary column (60m×0.25 mm, 0.25 um film thickness) was used for GC analysis. The

GC injector was set on splitless mode and operated at 60℃ initial temperature holding for 5 min, then heated up to 285 ℃ at 10 ℃/min holding for 15 min. This analysis method achieved good linearity (R>0.9993) and recoveries ranging from 80% to 100%. This study obtained 2528-15,070 $ng \cdot g^{-1}$ dw of D4, 2106-82,112 $ng \cdot g^{-1}$ dw of D5 and 1840-11,935 $ng \cdot g^{-1}$ dw of D6 in sludge samples. The concentrations of D5 and D6 in urban soil samples ranged from 11-30 $ng \cdot g^{-1}$ dw and 7.2-47 $ng \cdot g^{-1}$ dw, respectively. Concentrations of D4 in urban soil samples were under limit of detection (Companioni-Damas et al., 2012).

The analysis of siloxanes in water has been performed by Headspace-GC/MS method by Sparham et al., (2008). For this method, extraction was not necessary because siloxanes volatilize into the headspace and were pressurized through the inlet onto the GC coupled with MS. Ultrapure water and 20 mL headspace glass vials were used. First the sample was heated at 80 ℃ for 10 min for equilibrium. Then 3 mL of the headspace was injected via the GC injector which has a temperature at 250 ℃. After keeping the initial temperature at 40 ℃ for 4 min, the oven was heated to 200 ℃ at 8 $℃ \cdot min^{-1}$ and held for 5 min, before heating to 250 ℃ at 20 $℃ \cdot min^{-1}$. Finally the MS detector was operated using single ion monitoring. The internal standard was $[^{13}C_5]$decamethylcyclopentasiloxane ($^{13}C_5$–D_5). In this study, a non-siloxane-based column was used which is a polyethylene glycol- based stationary phase DB-Wax column, that gives a more accurate result. The detected concentration of D5 was <10-30.6 $ng \cdot L^{-1}$ in the river samples and 31-400 $ng \cdot L^{-1}$ in the treated wastewater samples (Sparham et al., 2008).

Companioni-Damas et al. (2011) conducted the analysis method to measure the concentrations of siloxanes also in water using headspace-solid phase microextraction (HS-SPME) and GC/MS. 20 mL water sample was spiked into a 40mL screw cap glass vial which was fitted with black Viton septa. The septa contained a stainless steel rod10 mm×5mm PTEF-coated stir bar. After 3 min vortex mixing and 10 min 25 ℃ water bath, the samples were extracted with a 65 μm-PDMS/DVB fiber at 25℃ for 40min using a constant magnetic agitation rate of 750 rpm. Then the fibers were exposed in the GC injector port at 240 ℃ for 5 min in order to reach thermal desorption. DB-5 MS fused silica capillary column (60 m ×0.25 mm I.D., 0.25 μm film thickness) was applied on GC. GC oven was held at 40 ◦C for 2 min before being heated to 250 ◦C at 10 ℃·min^{-1} and held for 5 min. Helium gas flow-rate was 1mL·min^{-1}. M4Q was the internal standard while the acetone was solvent used for the stock solution. This method obtained good linearity (R>0.999) and low limits of quantification ranging from 0.01 to 0.74 ng·L^{-1} for linear siloxanes and 18-34 ng·L^{-1} for cyclic siloxanes. This study reported 22.9 ng·L^{-1} and 58.5 ng·L^{-1} of D5 in two testing rivers and 21.2 ng·L^{-1} of D6 in one of them (Companioni-Damas et al., 2011).

The mass spectrometry is the most commonly used method for detecting siloxanes because it is used to characterize, identify and quantify compounds and it provides accurate results. The mass-to-charge ratios (m/z) of D4 and D5 are shown in following Table 2-6.

Table 2-6: Ion Selected for the Analysis of Siloxanes

Compound	Ions Selected (m/z)
D4	133, 281
D5	267, 355
M4Q	281, 369

Chapter 3. Problem Statement

3.1 General Statement

Due to the high energy requirements, an increasing number of wastewater treatment plants use the biogas to generate electricity and heat. The biogas is produced by the anaerobic digestion of the waste activated sludge. The anaerobic digestion not only solves the problem of reducing the volume of waste activated sludge, but also produces biogas for beneficial usage. The composition of biogas includes mainly methane (CH_4), carbon dioxide (CO_2), carbon monoxide (CO), hydrogen sulphide (H_2S), water and other impurities such as siloxanes. The occurrence of siloxanes, which are from the cosmetics and personal care products, will convert to silica scaling (abrasive microcrystalline silica) during the combustion of biogas. The silica scaling, which has very similar chemical and physical properties as glass, tends to attach to and accumulate on the inner surface of the boilers. This material leads to attrition of the gas engine surfaces and overheating of sensitive motor parts, which may cause serious major engine failure.

The scaling process reduces the efficiency of the energy generation. Also maintenance becomes an issue because it is expensive and time consuming to clean the engine and pipeline or to replace failed equipment. The available techniques to remove siloxane in the biogas are relatively expensive, resulting the higher cost of biogas. Efficient and accurate methods to measure the amount of siloxanes in wastewater and sludge have to be developed and validated in order to achieve better evaluation of the occurrence of siloxanes and removal procedures.

City of Loveland Wastewater Treatment Plant is one of the WWTPs that has been experiencing scaling issues due to occurrence of siloxanes in biogas. The plant has 29 square miles of service area with a peak wet hourly flow rate of 11.6 MGD. The average flow rate from April to

September is 6.2 MGD and during the rest of the year is 5.7 MGD. This plant operates anaerobic digestion to produce biogas for in-house heating. It was observed that the inner surfaces of the fire tubes were coated by the silica scaling after a few months. Figure 3-1 illustrates the accumulation of silica scaling on the fire tubes. The picture on left was captured in June 2010 while the right one was took in March 2011 (Kulkarni, H. V., 2012). As seen in Figure 3-2 same problem is observed by the Drake Wastewater Reclamation Facilities. These pictures were taken in 2009 during a cleaning event.

Figure 3-1: Effect of Silica Scaling on Fire Tube (Kulkarni, H. V., 2012)

Figure 3-2: Effect of Silica Scaling on Fire Tube in Drake WWRF (Photograph by Link Mueller)

3.2 Goal of This Study

The goal of this study was to optimize an analysis method and to determine the occurrence of siloxanes (D4 and D5) in wastewater and sludge samples from the City of Loveland WWTP and Drake WWRF, and river samples samples over time. The adopted and modified detection method was Headspace-Gas Chromatography/Mass spectrometry (HS-GC/MS) which is one of the most commonly used methods.

Chapter 4. Analysis of Octamethylcyclotetrasiloxane and Decamethylcyclopentasiloxane in Wastewater, Sludge and River Samples by Headspace Gas Chromatography/Mass spectrometry

4.1 Introduction

Siloxanes are widely used in various industrial processes and consumer products, such as detergents, shampoos, cosmetics, paper coatings and textiles (Dewil et al., 2006). They have been produced commercially since 1940s (Hunter et al., 1946). Two most commonly used cyclic siloxanes are Octamethylcyclotetrasiloxane (D4) and Decamethylcyclopentasiloxane (D5). The annual worldwide production of siloxanes is estimated at over one million tons (Hagmann et al., 1999). They have a low toxicity to humans, which makes them a common additive to the personal care products. After washing and showering, siloxanes from the personal care products are flushed into sewer pipelines and end up at the wastewater treatment plants (WWTPs). They are biodegradable in the atmosphere, but they are not degraded by the wastewater treatment processes. Because of their low water solubility, siloxanes accumulate in the waste activated sludge of the WWTPs and end up in the biogas if it is generated by anaerobic sludge digestion. During the combustion of the biogas, siloxanes are converted into abrasive microcrystalline silica, which has chemical and physical properties similar to those of glass, and form scales on the equipment. This residue leads to the abrasion of gas motor surfaces, overheating of sensitive motor parts and depresses the function of spark plugs leading to serious motor damage and shortening operation time (Appels et al., 2008).

Siloxanes were recognized as an issue in 1990s (Wang et al., 2013) and started to be measured in environmental samples, especially in wastewater and sludge. The average concentrations in

Swedish WWTPs' sludge samples were 0.4 $\mu g \cdot g^{-1}$ dry solid of D4 and 9.5 $\mu g \cdot g^{-1}$ dry solid of D5 (Kaj et al., 2005). Zhang et al. (2010) reported mean D4 and D5 concentrations at 0.1 $\mu g \cdot g^{-1}$ dry solid and 0.3 $\mu g \cdot g^{-1}$ dry solid respectively in sludge samples from a WWTP in northeastern China. 0.1 $\mu g \cdot g^{-1}$ dry solid of D4 and 15.1 $\mu g \cdot g^{-1}$ dry solid of D5 were detected in the sludge in WWTP in Greece (Bletsou et al., 2013). The occurrence of siloxanes in surface waters was also reported as they are discharged with the effluents from WWTP into the receiving water bodies. In two UK rivers D5 was detected at <0.01-0.03 $ug \cdot L^{-1}$ (Sparham et al., 2008). A study reported <0.01-0.02 $ug \cdot L^{-1}$ of D4, <0.03-1.48 $ug \cdot L^{-1}$ of D5 in rivers studied in Canada (Wang et al., 2012). D4 is very toxic to some aquatic organisms such as midge, daphnids and rainbow trout at very low concentrations of 6.3 $ug \cdot L^{-1}$ (European Commission, 2010, Kent et al., 1994, Sousa et al., 1995). D4 and D5 were not a serious concern for human health but they were defined harmful to the environment due to their potential to cause ecological harm (Environment Canada and Health Canada, 2008). They are also under consideration by UK Environment Agency and Canadian Environmental Assessment Agency for drinking water regulations. D4 was classified as Category 3 for reproductive toxicity by the European Commission.

This study focused on determination of D4 and D5 in wastewater and sludge samples from two local WWTP, and river samples by Headspace-Gas Chromatography/Mass spectrometry (HS-GC/MS) analysis.

4.2 Materials and Methods

4.2.1 Chemicals and glassware

The standard solutions of Octamethylcyclotetrasiloxane (D4) and Decamethylcyclopentasiloxane (D5) were purchased from TCI-America (Portland, OR) at 98.0 % and 97.0 % purity, respectively. Tetrakis(trimethylsiloxy)siloxane (M4Q) at 98% purity was purchased from Alfa Aesar (Ward Hill, MA). Acetone (99.7%) was purchased from Fisher Scientific (Pittsburgh, PA). They were all stored at room temperature in the dark except M4Q which was stored at 4 ℃ in dark.

The glass syringes at 50 µL, 250 µL, 1 mL and 10 mL were purchased from Fisher Scientific (Pittsburgh, PA). 20 mL headspace vials with flat bottoms and butyl/PTFE aluminum crimp caps (20 mm) were purchased from Agilent (Santa Clara, CA). The vials were used without any pretreatment. 1 L beakers, 500 mL flasks and 40 mL amber glass vials with screw caps were also purchased from Fisher Scientific (Pittsburgh, PA). The cap-fitted septa with diameter of 22 mm were purchased from Thomas Scientific (Rockwood, TN). Pierce hand crimper (Rockford, IL) was used to cap the Headspace vials. An Analog Vortex Mixer (No. 02215365) from Fisher Scientific (Pittsburgh, PA) was used to homogenize the samples. The disposable aluminum dishes were obtained from Fisher Scientific (Pittsburgh, PA) for the total solids determination.

4.2.2 Stock Solutions and Calibrations Curves

D4 and D5 stock solutions were prepared fresh daily in acetone for the calibration curve samples at 2 concentrations of 10 $mg \cdot mL^{-1}$ and 42.5 $\mu g \cdot mL^{-1}$. The secondary stock solutions were prepared by successive dilution of the original stock solutions with acetone at 6 concentrations of 17 $\mu g \cdot mL^{-1}$, 3.4 $\mu g \cdot mL^{-1}$, 1.7 $\mu g \cdot mL^{-1}$, 340 $ng \cdot mL^{-1}$, 170 $ng \cdot mL^{-1}$ and 34 $ng \cdot mL^{-1}$. M4Q stock solution was prepared in acetone at 2 µg/mL to be used as the internal standard. It was diluted to 136 $ng \cdot mL^{-1}$ to get the secondary stock solution. Then 0.5 mL of the secondary stock solution was spiked in 19.5 mL of water to achieve the final concentration of 3.4 $ng \cdot mL^{-1}$ in the final stock solution. All of these solutions were prepared in amber glass vials with Teflon line caps.

The calibration curve samples were prepared in sludge and distilled water samples at 0.5 $ng \cdot mL^{-1}$, 1 $ng \cdot mL^{-1}$, 5 $ng \cdot mL^{-1}$ and 15 $ng \cdot mL^{-1}$ of D4, 0.5 $ng \cdot mL^{-1}$, 5 $ng \cdot mL^{-1}$, 50 $ng \cdot mL^{-1}$ and 150 $ng \cdot mL^{-1}$ of D5, 0.25 $ng \cdot mL^{-1}$ of M4Q.

4.2.3 Sample Collection and Preparation

The wastewater and activated sludge samples were collected from City of Loveland Wastewater Treatment Plant (WWTP), Loveland and Drake Wastewater Reclamation Facility (WWRF), Fort Collins CO, from November 2013 to May 2014. Loveland WWTP has an average flow rate of 6.2 MGD during April to September and 5.7 MGD during the rest of the year (City of Loveland, 2013), while the Drake WWRF has an average flow rate of 11.2 MGD during April to September and 10.2 MGD during the rest of the year. Both of the plants use activated sludge process to treat wastewater. The samples obtained were: influent, primary sludge, primary effluent, return activated sludge, centrate, digester sludge and effluent. The water samples of Big Thompson River and Fossil Creek Ditch were collected at 1m, 10 m and 50 m from the discharging points in April, 2014. All of the samples were collected in amber glass bottles without headspace and chilled during transportation, and stored in the dark at 4 ℃ until sample preparation and analysis.

The sludge and wastewater samples were brought to room temperature for one hour. The sludge calibration curve standards were prepared as follows: 1.5 mL of waste activated sludge was placed in the headspace vial and received 1 mL of reverse osmosis (RO) water and 0.5 mL of internal standard water solution (1.5 $ng \cdot mL^{-1}$). The sludge calibration curve standards were prepared the same way, with an addition of 25 µL of corresponding D4, D5 stock to the headspace vial to achieve concentrations of 0.5 $ng \cdot mL^{-1}$, 1 $ng \cdot mL^{-1}$, 5 $ng \cdot mL^{-1}$, 15 $ng \cdot mL^{-1}$ of D4 and 0.5 $ng \cdot mL^{-1}$, 5 $ng \cdot mL^{-1}$, 50 $ng \cdot mL^{-1}$, 150 $ng \cdot mL^{-1}$ of D5. Finally, the vials were sealed with a crimped septa cap. The sludge samples were prepared as the same manner as the sludge calibration curve standards except by adding 50 μL of pure acetone instead. The wastewater calibration curve standards were prepared as follows: the headspace vials received 2.5 mL of RO water, 0.5 mL of internal standard water solution (1.5 $ng·mL^{-1}$), 25 μL each of corresponding D4, D5 stock solutions with the needle just under the liquid surface to make the same headspace concentrations as the sludge standards. The wastewater samples were prepared the same way as the wastewater calibration curve standards except by adding 50 μL of pure acetone instead. Both the sludge and water blanks were also prepared in the same manner as the corresponding sludge calibration curve standards and wastewater calibration curve standards with added 50 μL of pure acetone instead to even out the volume. The concentration of M4Q in all the samples was 0.25 $ng·mL^{-1}$. All the samples were tightly capped by a crimper. Then all the samples were well homogenized by the Vortex Mixer for 3 min each. Each sample was prepared in duplicates.

4.2.4 Method Validation

The following parameters, included specificity, selectivity, intermediate precision, matrix effects, linearity, limit of detection (LOD), and recovery were determined to validate this analysis method (Huber, 2010). This procedure was performed by spiking known concentrations of D4 and D5 to blank samples (RO water and sludge), with the exception of the linearity. Triplicated samples with the same concentration was analyzed and compared to each other while the analysis was repeated for three days (Nov 2, Nov 3 and Nov 4, 2013). The concentrations series were prepared at 0, 0.1, 0.5, 1, 5, 10, 50 $ng•mL^{-1}$ of both D4 and D5.Then every sample received 0.1 $ng•mL^{-1}$ of M4Q. For wastewater samples, 2.5 mL of RO water and 0.5 mL of M4Q water solution were added into headspace vial. For sludge samples, 1.5 mL of waste activated sludge from Lovelan WWTP, 1.0 mL of RO water and 0.5 mL of M4Q water solution were spiked into the vial. Every sample were added 50 μL of the corresponding D4 and D5 mixture stock solution except the blanks received 50 μL of pure acetone. The recovery was determined by calculating the ratio between the samples spiked known concentrations and the calculated concentration from the calibration curve equation. The spiked concentrations were 0.25, 2.5, 7.5 ng•mL^{-1} in both wastewater and sludge samples. Tiplicates were prepared. The HS-GC/MS analysis was conducted as the same procedure as the samples testing and described in the following section.

4.2.5 HS-GC/MS Analysis

The Gas Chromatography and Mass spectrometry analysis was performed by a Waters Quattro Micro GC/MS system. This analysis was adopted and modified from a study done by Sparham et al., (2008).The carrier gas was ultra-high purity helium at a head column pressure of 79 kPa and flow rate at 1 mL·min^{-1}. The GC column, J&B DB-WAX (30 m ×0.25 mm, 0.5 μm film thickness) was purchased from Agilent. The Headspace sampler is Hewlett-Packard (HP) 7694. The headspace vials were placed in the headspace oven to heat and equilibrate for 20 min at 85℃. (Other parameters: transfer line 95℃, loop 105 ℃, injection time 1 min, loop equilibration time 0.01 min, loop fill 0.2 min, pressurization time 0.15 min). 2 mL of headspace was injected into the GC column with a split ratio of 1:10. The oven of GC (Agilent 6890) was held at an initial temperature of 75℃, then to 100 ℃ at 5 ℃/min, and finally to 200 ℃ at 120 ℃/min. The MS source temperature and GC interface temperature were 220 ℃ and 280℃, respectively. The mass spectrometer (Waters Quattro MicroTipleQuad) was operated in positive electron ionization mode with selective ion monitoring for each analyte and internal standard with a dwell time of 0.1s. The electron energy and electron energy were 70 eV and 200 μA, respectively. The ions monitored were 193 and 281 m/z for D4, 267 and 355 m/z for D5 and 281 and 369 m/z for M4Q. MassLynx v. 4.1 software was used for data acquiring, integrating and processing.

4.2.6 Total Solids Analysis

Total solids analysis was performed for the sludge samples to report the D4 and D5 per mass of dry solids. The sludge samples (primary sludge, RAS, TWAS and digester sludge) were vortex mixed and 10 mL of each sludge sample was pipetted into a disposable aluminum dish and were dried at 105℃ for 24 h. Then the samples were cooled to room temperature in a desiccator, and the samples were weighed. The total solids concentration was determined by the ratio between the dried mass and the wet volume.

4.3 Results and Discussions

4.3.1 Method validation

4.3.1.1 Specificity

The sludge samples spiked with D4 and D5 were used to determine the specificity and selectivity. To identify each siloxane analyzed, a certain retention time was observed within ±1s (±0.5-0.7%). This tolerance is smaller than that in the study done by Sanchis et al. (2013) which had a retention time tolerance of ±2.5%. The ion ratios of 2 selected ions for each siloxane were checked and confirmed to stay in the same range. (D4: 281, 133 m/z; D5: 355, 267 m/z; M4Q: 281, 369 m/z). Table 4-1 shows the retention times and ion ratios for siloxanes analyzed. In the complicated sludge matrix, this method was able to identify and analyze characteristic siloxane ions selected qualitatively. Figure 4-1 shows the isolated sharp peaks and retention times for siloxanes analyzed in the sludge matrix.

Table 4-1: Retention Times and Ion Ratio

Compound	Retention Time (min)	Ion Ratio
D4	2.38	5.00 ± 0.42
D5	3.24	0.876 ± 0.024
M4Q	3.38	5.64 ± 0.13

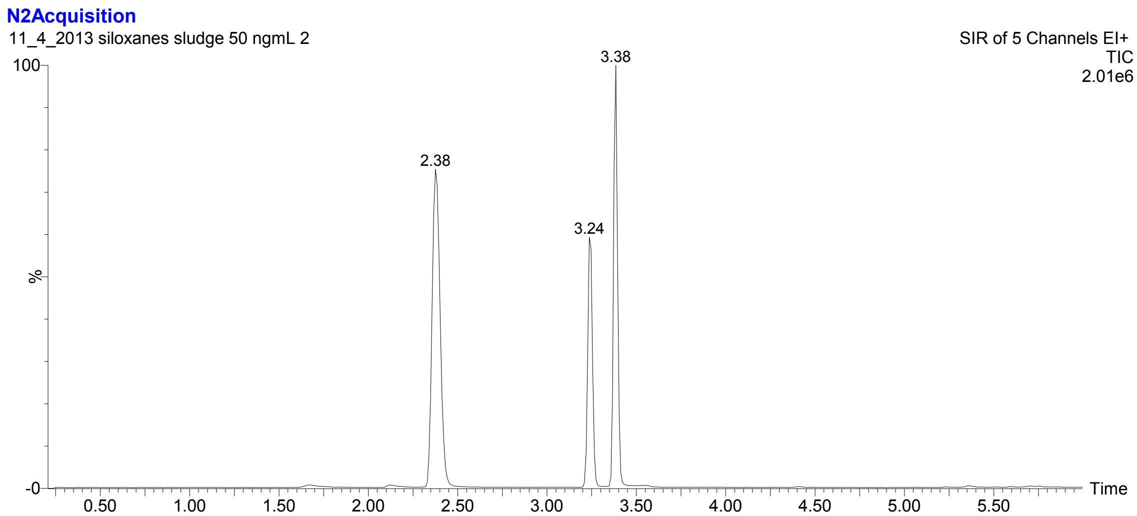


Figure 4-1: Peaks and Retention Time

4.3.1.2 Linearity

The linearity was determined by performing a 6-points calibration curve for D4 and D5 in wastewater and sludge standard samples. Calibration curve standard samples were prepared by spiking known concentrations in the wastewater and sludge samples. All the peak areas of wastewater samples were corrected by subtracting the peak areas of the blank wastewater samples while the ones of sludge samples were correceted by deleting the ones of the blank sludge samples. Then these samples were analyzed. It was observed that good linearity was showed by least squared regression (r^2>0.9896). Table 4-2 shows the summary of the correlation coefficient (r^2). In comparison, r^2 =0.9896 of calibration curve for D5 in wastewater tested on Day 1 was slightly lower than other r^2 (r^2> 0.990).

Table 4-2: Correlation Coefficient of Calibration Curves

		Day 1	Day 2	Day 3	Mean±Stdv
D4	Wastewater	0.9960	0.9969	0.9981	0.9970±0.0011
	Sludge	0.9961	0.9950	0.9990	0.9967±0.0021
D5	Wastewater	0.9896	0.9961	0.9936	0.9931±0.0033
	Sludge	0.9982	0.9968	0.9951	0.9967±0.0016

4.3.1.3 Repeatability

The repeatability of this method was determined by running triplicates of water and wastewater samples spiked with M4Q at 0.1 ng·mL^{-1} and following the same procedure for three consequtive days based on the guidelines of United Nations Office on Drugs and Crime, (2009) Table 4-3 shows the average peak areas and standard deviation of areas and the coefficients of variation of the internal standard. The average coefficients of variation for each day are as follows: 6.07% for Day 1, 7.85% for Day 2 and 8.43% for Day 3, which were under the requirement of validation of analytical methodology by United Nations Office on Drugs and Crime (below 20%) (United Nations Office on Drugs and Crime, 2009).

Table 4-3: Average Peak Areas, Standard Deviation of Areas and Coefficients of Variation of M4Q

	Concentrations of D4 and D5 (ng·mL^{-1})	Average Peak Area			Standard Deviation			Coefficient of Variation (%)		
		Day 1	Day 2	Day 3	Day 1	Day 2	Day 3	Day 1	Day 2	Day 3
Water Calibration Curve Samples	0	11970	15894	16391	930	1101	3874	7.77	6.93	23.63
	0.1	13206	16458	23561	680	787	2140	5.15	4.78	9.08
	0.5	12950	16409	22312	718	578	519	5.54	3.52	2.33
	1	13039	16291	21773	924	771	633	7.08	4.73	2.91
	5	13390	17733	22577	866	1519	1169	6.47	8.56	5.18
	10	13055	17963	20326	816	1415	1652	6.25	7.88	8.13
	50	12839	18144	21483	1112	1542	181	8.66	8.50	0.84
Sludge Calibration Curve Samples	0	49789	45922	33077	4019	2504	9104	8.07	5.45	27.52
	0.1	54214	45331	36867	3374	5514	3682	6.22	12.16	9.99
	0.5	51949	40831	37002	6422	8129	1643	12.36	19.91	4.44
	1	56552	46867	37447	2981	5083	941	5.27	10.85	2.51
	5	58876	51668	37468	105	2511	1365	0.18	4.86	3.64
	10	57894	48349	38640	1279	2200	1203	2.21	4.55	3.11
	50	57141	49855	40967	2118	3580	6027	3.71	7.18	14.71

Tables 4-4 and 4-5 illustrate the coefficients of variation (CV) of target siloxanes in wastewater and sludge standard samples on three consequtive days. The CV of sludge standards was smaller than that of wastewater standards in general. The CV of sludge standards ranged from 1.1% to 17.9% while that of wastewater standards were in the range of 1.6%-37.2%. The largest CV values were observed as 27.2% of CV of D4 and 37.2 % of CV of D5 both in wastewater standard samples on Day 1. The average CV of 3-days were 15.4% for D4 in wastewater, 7.4% for D4 in sludge, 14.5% for D5 in wastewater and 9.2% for D5 in sludge. It was reported by Wang et al, (2013) that the mean CV in the analysis were 21% for D4 and D5 in wastewater samples while 8% for D4 and 6% for D5 in biosolid samples. In the study done by Bletsou et al, (2013), the CV was reported as 29% for D4 and <17% for D5 in water samples and <12% for both siloxanes in sludge samples. In this study, the mean CV in wastewater samples was much smaller and the one in sludge samples was similar compared to the discussed researches. This study exhibits good reliability for analysis. Based on these findings it was concluded that this method is repeatable, which will ensure the quality of the analysis.

Table 4-4: Coefficients of Variation (%) of D4 in Wastewater and Sludge Standard

	Concentration ($ng \cdot mL^{-1}$)	Day 1	Day 2	Day 3
Wastewater Standard	0.1	21.4	17.5	12.4
	0.5	8.1	18.1	14.0
	1	20.4	22.8	9.4
	5	27.4	14.8	17.8
	10	8.1	21.8	9.8
	50	18.7	7.5	7.6
	Average	17.3	17.1	11.8
Sludge Standard	0.1	6.5	7.1	16.1
	0.5	1.8	10.8	8.1
	1	2.9	2.9	7.8
	5	10.6	4.9	10.0
	10	2.4	7.2	4.0
	50	9.3	3.2	17.9
	Average	5.6	6.0	10.7

Table 4-5: Coefficients of Variation (%) of D5 in Wastewater and Sludge Standard

	Concentration ($ng \cdot mL^{-1}$)	11/2/2013	11/3/2013	11/4/2013
Wastewater Standard	0.1	20.1	3.2	13.6
	0.5	1.6	9.4	11.0
	1	9.7	15.2	3.3
	5	37.2	13.8	15.6
	10	16.1	27.2	7.7
	50	33.2	16.7	5.6
	Average	19.7	14.3	9.5
Sludge Standard	0.1	15.2	12.5	12.4
	0.5	7.1	7.3	13.9
	1	9.4	6.9	5.3
	5	12.3	3.8	12.4
	10	10.9	6.7	9.0
	50	11.3	8.7	1.1
	Average	11.0	7.7	9.0

4.3.1.4 Intermediate Precision

The intermediate precision represents the variations on different days (Huber, 2010). As shown in Table 4-3, it was observed that the average areas of internal standard (M4Q) in the water standards increased over the 3 testing days. There was an average of 31% rise from Day 1 to Day 2 and an average of 25% from Day 2 to Day 3 among the water calibration curve samples. However, the M4Q areas were decreased over the 3 days in the sludge calibration curve samples: an average of 15% from Day 1 to Day 2, an average of 20% from Day 2 to Day 3. This was due to the complicated composition of sludge matrix that lowered the MS sensitivity, and that impacted the next analysis. The measurement was conducted in the same day ensuring the accurateness of the analysis.

4.3.1.5 Limit of Detection

The limit of detection (LOD) is "the lowest amount of analyte in a sample which can be detected but not necessarily quantitated as an exact value" (Huber, 2010). It is usually defined as the concentrations of analyte occupy a peak area as three times higher as that of blank noise level (Sanchis et al., 2013, Companioni-Damas et al., 2012, United Nations Office on Drugs and Crime, 2009). The wastewater blanks with pure acetone were prepared the same as the other blanks. After GC/MS analysis, the average peak areas of D4 and D5 in the blanks were selected as the noise level for LOD detection. This GC/MS analysis has LODs as follows: 0.22 ng/mL of D4 and 0.31 ng/mL of D5.

4.3.1.6 Recovery

Sludge and wastewater samples, which were from the same origin were used for the calibration curve standards, were prepared for the recovery test. Reported in Table 4-6, the recoveries of 94±4% and 98±6% were achieved for D4 and D5 in wastewater samples respectively, while recoveries of 125±15% were obtained for D4 spiked sludge samples and 63±24% for D5. Compared to another study using HS-GC/MS analysis method, a greater recovery of D4 and a lower recovery of D5 were obtained in the sludge samples in this study. Wang et al. (2012) conducted a large-volume injection-gas chromatography/mass spectrometry analysis method to test siloxanes and achieved recoveries of 92±15% of D4 and 114±32% of D5 in sludge samples. Recoveries of 80±4% for D4 and 85±5% for D5 in wastewater samples were reported in the study with HS-GC/MS analysis by van Egmond et al, (2013). The recoveries were larger in this study than that in the study done by van Egmond et al, (2013).

Table 4-6: Recovery of D4 and D5 in Wastewater and Sludge Samples (Mean ± Standard Deviation)

Recovery (%)	D4	D5
Wastewater Samples	92±4	98±6
Sludge Samples	125±15	63±24

4.3.1.7 Matrix Effects

There were considerable differences between water and sludge matrices. Even though the same mass of D4 and D5 were added to water and sludge samples, the peak areas in the water samples were higher than the sludge samples. The differences between the slopes of calibration curve equations show the matrix effects indirectlyTable 4-7 shows the comparisons between slopes of water and sludge matrix over 3 days by determining the linear regression equations with all $R^2>0.99$ except the one for D5 in wastewater on Day 1 ($R^2=0.9896$). The slopes of wastewater calibration curve samples were significantly larger than that of sludge ones. Slopes of D4 in both sample types were higher than that of D5. The matrix effects were observed due to the low water solubilities of siloxanes and likely absorption or adsorption by the organic matter in the activated sludge.

Table 4-7: Slopes of Linear Regression Equations between Concentrations and Peak Areas

Slopes	D4			D5		
	Day 1	Day 2	Day 3	Day 1	Day 2	Day 3
Wastewater Calibration Curve Samples	7965.3	6437.5	5154.6	1274.7	986.8	983.5
Sludge Calibration Curve Samples	2556.7	2264.6	1424.7	382.5	338.2	191.5

4.3.1.8 Volume of Solvent Effects

The amount of acetone added in the sample may impact the amount of siloxanes volatilizing into headspace and going into GC column. Table 4-8 shows the difference in peak areas with the corresponding solution volume added. The first two samples received 550 and 775 ul of acetone solution, while the last two received 9 and 22.5 uL of acetone solution. The peak areas for both D4 and D5 were greater in the first two samples compared to the last two. As a result, it may be concluded that solvent-acetone impacts the volatility and solubility of siloxanes in the sludge samples and causes them to volatilize from the matrix.

Table 4-8: Peak Areas of Different Acetone Volume Added

Target Concentration ($ng{\cdot}mL^{-1}$)	Added Solution Concentration ($\mu g{\cdot}mL^{-1}$)	Added solution volume (μL)	Peak Areas	
			D4	D5
50	1	550	11423.5	32010.4
75	1	775	27156.2	42067.8
100	100	9	607.9	1731.1
250	100	22.5	3485.6	2427.2

Figures 4-2, 4-3 and 4-4 illustrate the impacts of acetone volume in the samples on the peak areas of D4, D5 and M4Q. 50 μL, 100 μL, 500 μL, 1000 μL and 1500 μL of acetone were added into the water and sludge samples with the same amount of D4, D5 and M4Q (1 $ng{\cdot}mL^{-1}$ D4, 5 $ng{\cdot}mL^{-1}$ D5 and 0.25 $ng{\cdot}mL^{-1}$ M4Q). With the acetone volume increasing to 1000 μL, the peak areas of D4, D5 and M4Q in water samples showed a raising trend except for the sample that received 1500 µL of acetone. The peak areas of D4, D5 and M4Q increased in both water and sludge samples with increasing volume of acetone.

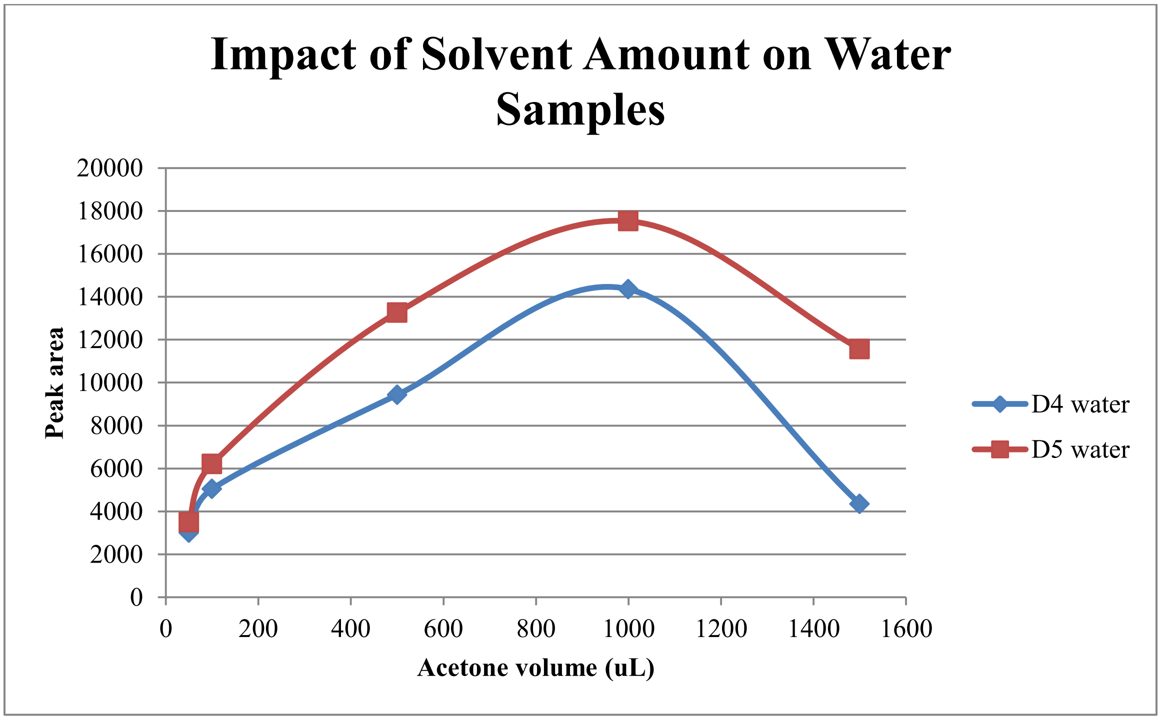


Figure 4-2: Impact of Solvent Amount on Water Samples Peak Areas

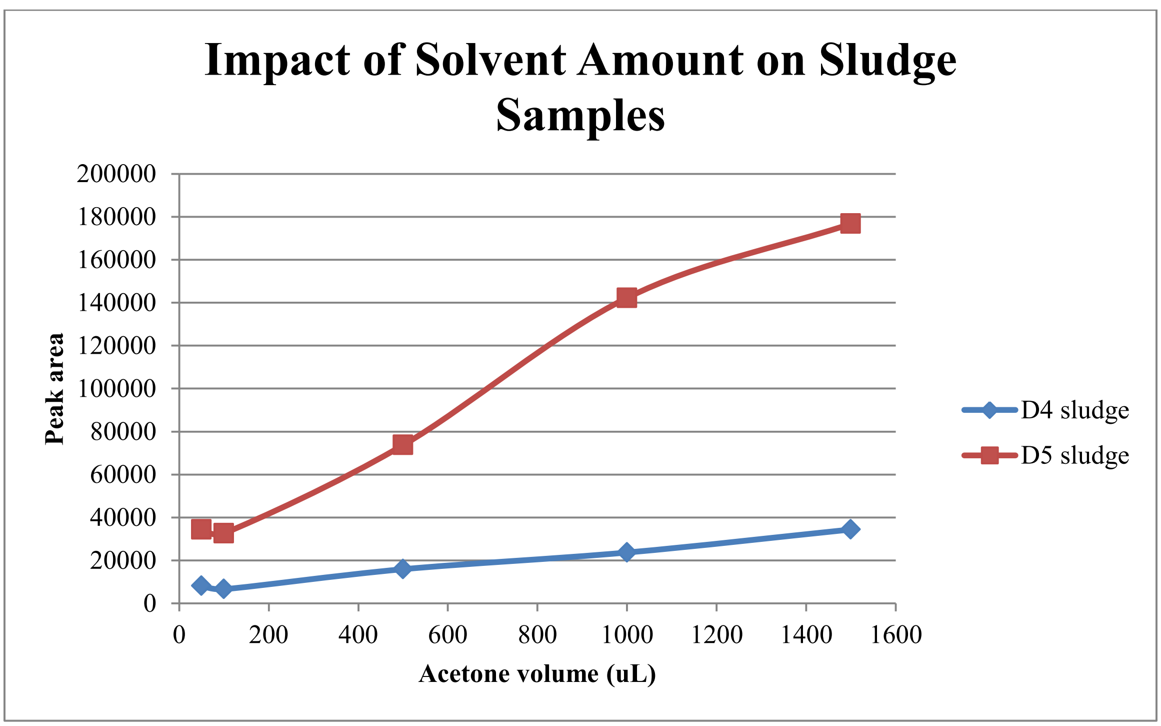


Figure 4-3: Impact of Solvent Amount on Sludge Samples Peak Areas

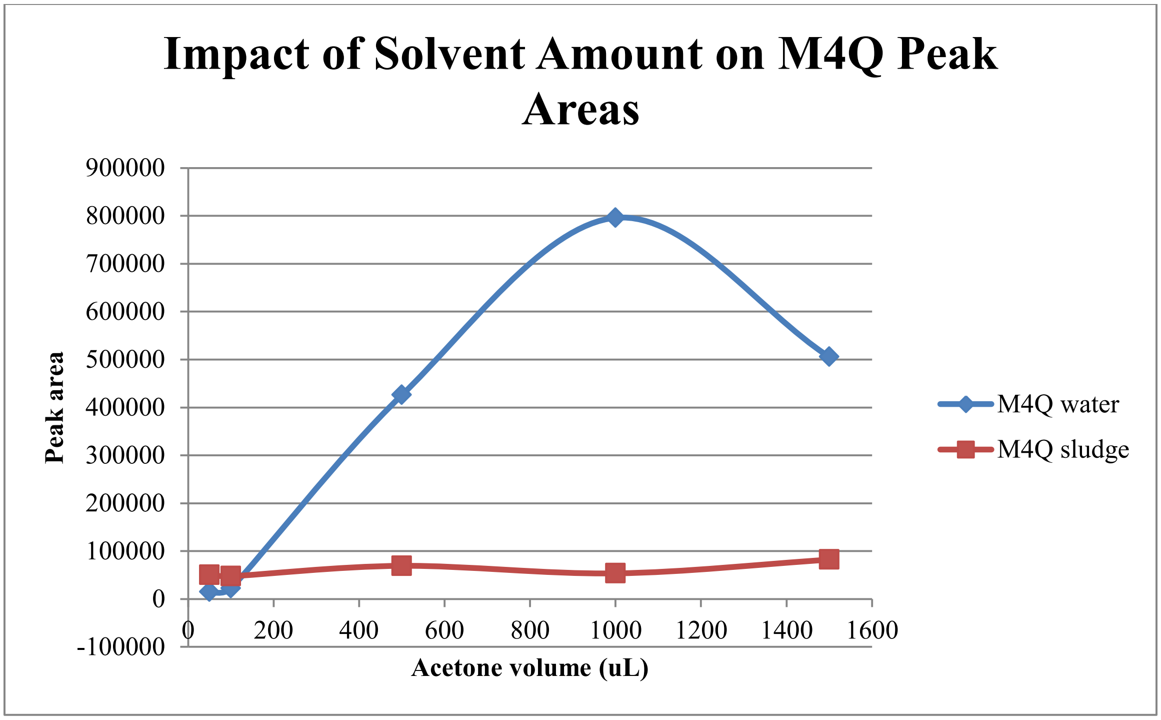


Figure 4-4: Impact of Solvent Amount on M4Q Peak Areas

4.3.2 Method Comparison

Sparham et al. (2008) measured D5 in river water and WWTP effluent by HS-GC/MS method. The isotope $^{13}C_5$-D5 was used as the internal standard (m/z 360). There are some other differences between this study and Sparham et al (2008) method. The HS conditions in Sparham et al. (2008) were as follows: Oven equilibrium temperature 80 °C, Transfer line temperature 120°C, Loop temperature 100°C, oven equilibrium time 10 min, injection time 1 min, loop equilibrium time 0.01 min, loop fill time 0.2min, pressurization time 0.15 min. The GC temperature ramp was as follows: the oven was set at 40°C initially and held for 4 min; then it was heating up to 220 °C at 8 °C/min and held for 5 min; finally it was heating up to 250 °C at 20 °C/min. There are the same parameters. Acetone was applied as the solvent for the stock solution in both methods. The J&W DB-Wax capillary column was used. The GC split ratio is 1:10. The MS detector was operated in EI mode using single ion monitoring.

Xu et al. (2013) measured D3, D4, D5 and D6 with the method of HS-SPME-GC/MS for the water and sludge samples from a WWTP in China. The M4Q was used as the internal standard because it is similar in chemical composition to siloxanes, not commonly present in environmental matrices and does not interfere with siloxanes. The 65 μm polydimethylsiloxane/diviylbenzene (PDMS/DVB) fiber was used as the extraction fiber, while the HP-5ms (30m×0.25mm I.D., 0.25μm film thickness) capillary column was used for separation. The GC was operated on splitless mode and the MS was operated on EI and SIM mode (Xu et al., 2013).

In this study, the application of headspace oven decreased the operation time of GC. It separated siloxanes from the sample matrix. The run time of GC was 5.83min per sample compared to 22.3 min in Kulkarni (2012) analysis, 33min in Sparham et al (2008) analysis and 38 min in Zhang et al (2009) analysis.

4.3.3 Results of Total Solids of Sludge

Figures 4-5 and 4-6 show the total solids concentrations (TS) of the sludge samples from Loveland Wastewater Treatment Plant and Drake Wastewater Reclamation Facility respectively on 5 testing days (January 16, February 5, March 6,April 15 and May 13, 2014). The return activated sludge (RAS) contained the lowest TS compared to other sludge samples for both plants. The primary sludge TS was higher than other samples. The thickened waste activated sludge (TWAS), which was the dewatered waste activated sludge, had higher TS compared to the anaerobic digester sludge.

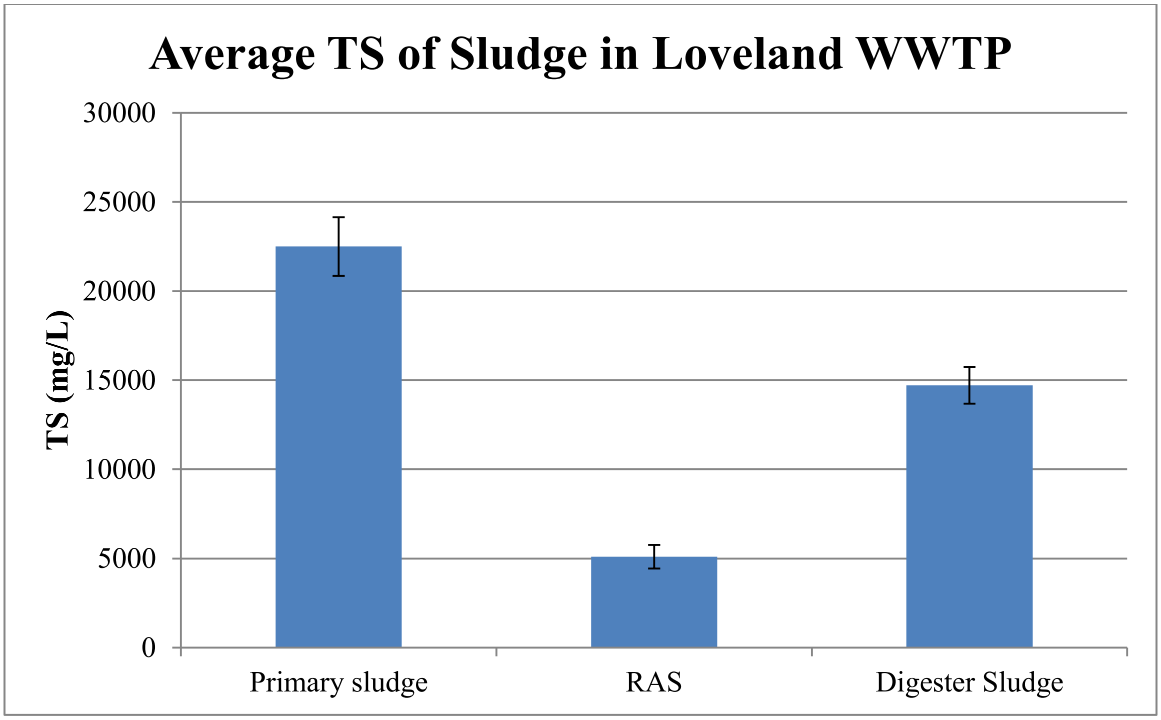


Figure 4-5: Average TS of Sludge in Loveland WWTP

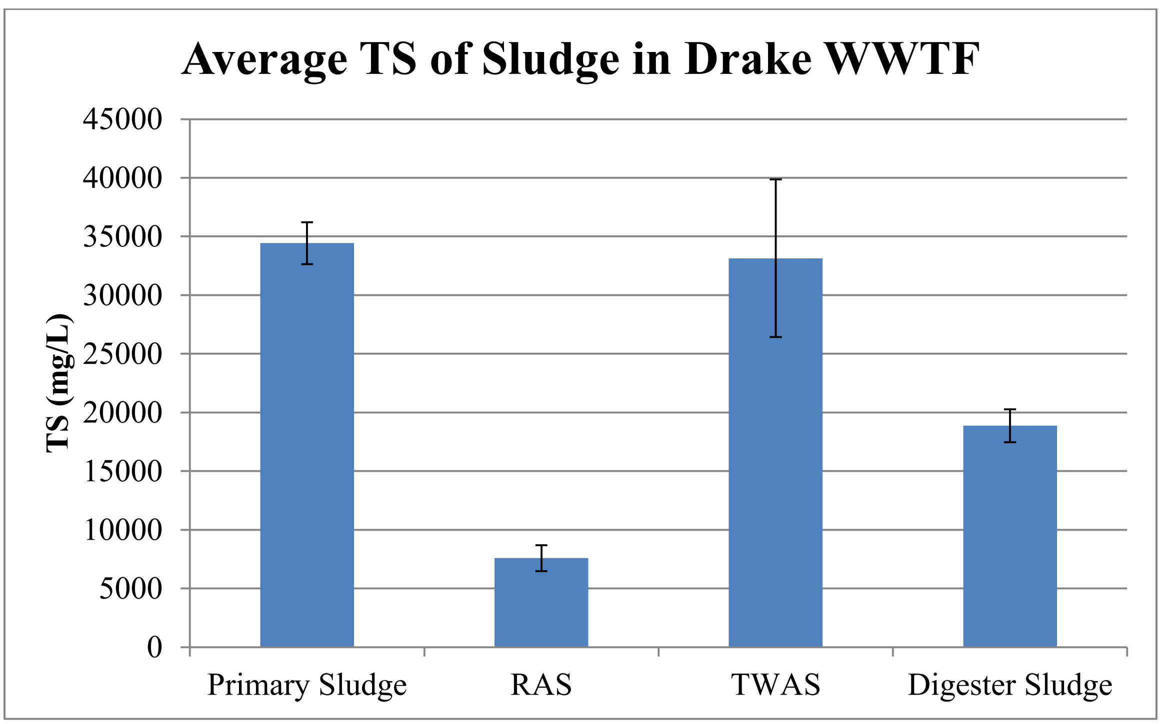


Figure 4-6: Average TS of Sludge in Drake WWRF

4.3.4 Results of Drake Wastewater Reclamation Facility, Fort Collins, CO

Siloxane concentrations from Drake WWRF were measured over 5 months as shown in Figures 4-7 and 4-8. D5 was detected at higher concentrations than D4 in the samples. The average concentrations of both D4 and D5 in effluent, influent and centrate were reported lower than the sludge samples, as expected as the siloxanes accumulate in the sludge. The siloxanes concentrations in sludge samples increased from November 2013 to January 2014, decreased in February 2014, increased from March to April 2014. The lowest concentration of D4 was obtained at 0.7 $ng \cdot mL^{-1}$ in effluent sample on January 13, 2014 while the highest one at 73.7 $ng \cdot mL^{-1}$ in thicken waste activated sludge (TWAS) on the same day. The lowest concentration of D5 was observed at 0.4 $ng \cdot mL^{-1}$ in effluent sample on March 7, 2014 while the highest one at 1075.2 $ng \cdot mL^{-1}$ in thicken waste activated sludge on January 13, 2014. The differences between the siloxanes levels in thicken waste activated sludge and anaerobic sludge (sludge from anaerobic digester) yield the amount of siloxanes that volatilized and ended up in the biogas. The average concentrations in influent were larger than that in effluent indicating that there is some removal of siloxanes in the treatment process. The product of concentration and flow rate determined the mass loadings of siloxanes. Shown in Table 4-9, the mean daily mass rates of D4 and D5 in effluent was reduced compared to that in the influent. 53% of D4 and 59% of D5 was removed by the Drake WWRF. The siloxanes may be absorbed or adsorbed by the sludge or volatized into the atmosphere partially.

Table 4-9: Mean Mass Rate of D4 and D5 in Drake WWRF

	Influent	Primary Sludge	TWAS	Effluent
Flow Rate (MGD)	9.6	0.05	0.16	9
D4 ($g \cdot d^{-1}$)	159.9	4.4	16.1	74.9
D5 ($g \cdot d^{-1}$)	225.3	28.8	182.7	92.0

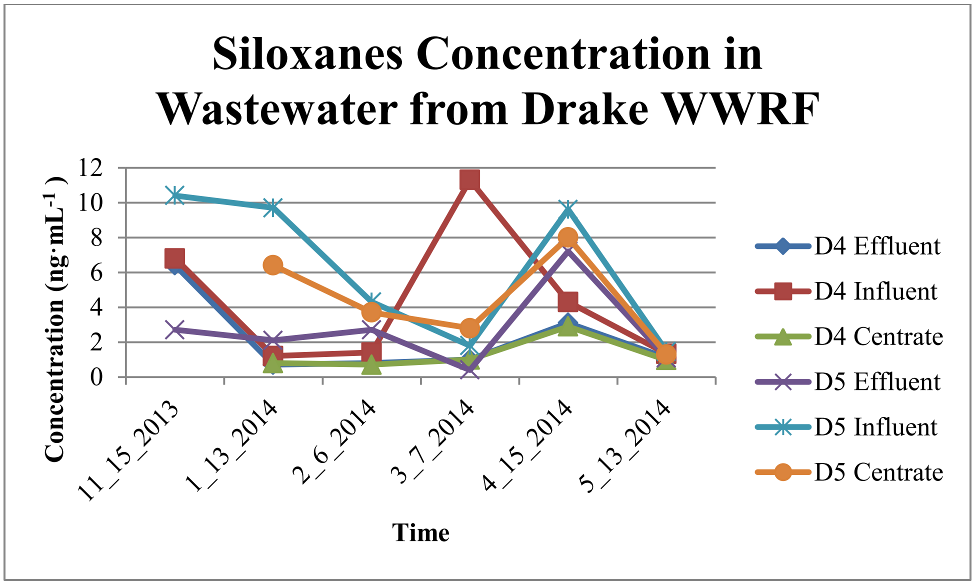


Figure 4-7: Concentrations of D4 and D5 in Wastewater samples in Drake WWRF

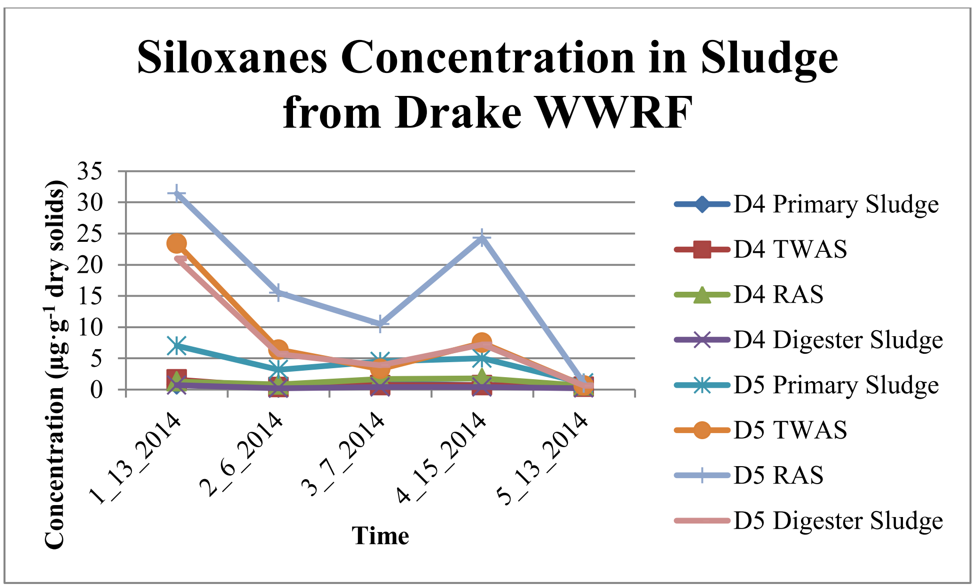


Figure 4-8: Concentrations of D4 and D5 in Sludge samples in Drake WWRF

Table 4-10 provides the concentrations of siloxanes in the sludge samples in the Drake WWRF as $\mu g \cdot g^{-1}$ dry solid. The D5 concentrations were greater than D4 in every sample due to the higher addition of D5 to personal care products compared to other siloxanes. The largest concentration of D4 was observed at 1.67 $\mu g \cdot g^{-1}$ dry solid in the return activated sludge (RAS) on March 7, 2014 while the smallest was at 0.32 $\mu g \cdot g^{-1}$ dry solid solid in primary sludge on

February 6, 2014. Then the largest concentration of D5 was obtained at 31.41 $\mu g \cdot g^{-1}$ dry solid in RAS on January 13, 2014 and the smallest was at 3.18 $\mu g \cdot g^{-1}$ dry solid in primary sludge on February 6, 2014. The concentrations of both siloxanes dropped from January to February and increased in April 2014. The digester sludge obtained the smallest concentrations of D4 compared to other sludge samples. The concentrations of D4 varied in a range 0.2-0.7 $\mu g \cdot g^{-1}$ dry solid in the digester sludge. The concentrations of D5 ranged from 3.9-21.0 $\mu g \cdot g^{-1}$ dry solid in the digester sludge.

Table 4-10: Concentrations of Siloxanes in Sludge Samples in Drake WWRF

Date			Primary Sludge	TWAS	RAS	Digester Sludge
1_13_2014	TS ($mg \cdot L^{-1}$)		49690	45910	9970	18005
	Concentration ($\mu g \cdot g^{-1}$ dry solid)	D4	0.8	1.6	1.3	0.7
		D5	7	23.4	31.4	21
2_6_2014	TS ($mg \cdot L^{-1}$)		33325	30495	8620	17570
	Concentration ($\mu g \cdot g^{-1}$ dry solid)	D4	0.3	0.4	0.8	0.2
		D5	3.2	6.4	15.5	5.7
3_7_2014	TS ($mg \cdot L^{-1}$)		30930	36635	7735	20755
	Concentration ($\mu g \cdot g^{-1}$ dry solid)	D4	1	0.7	1.7	0.4
		D5	4.5	3.3	10.5	3.9
4_15_2014	TS ($mg \cdot L^{-1}$)		34430	33145	7585	18870
	Concentration ($\mu g \cdot g^{-1}$ dry solid)	D4	0.7	0.7	1.8	0.4
		D5	5	7.5	24.3	7.2
5_13_2014	TS ($mg \cdot L^{-1}$)		16630	34185	8070	16060
	Concentration ($\mu g \cdot g^{-1}$ dry solid)	D4	0.7	0.5	0.6	0.2
		D5	1.1	0.6	1	0.6

4.3.5 Results of City of Loveland Wastewater Treatment Plant, Loveland, CO

Figures 4-9 and 4-10, show the concentrations of D4 and D5 in the wastewater and sludge samples obtained from Loveland WWTP.The concentrations of D4 and D5 in the influent on November, 15, 2013 were higher than that on the other testing days, which showed more siloxanes were discharged into the wastewater in November 2013 compared to the first four months of 2014. Among the water samples, concentrations of both D4 and D5 in the effluent samples were determined to be the smallest on the same testing days except D5 on April 15, 2014. Table 4-11 shows the mean mass loadings of the both siloxanes in Loveland WWTP. 4% of D4 and 58% of D5 removal was observed in Loveland WWTP. Because of circulation of return activated sludge, the siloxanes accumulate in the secondary treatment basin, which increased the amount of mass of siloxanes in the waste activated sludge. The concentrations of both siloxanes in digester sludge increased from November 2013 to January 2014, dropped in February 2014 and increased again in March 2014. The concentrations of D4 and D5 in effluent decreased from November 2013 to March 2014 and increased in April 2014. Among sludge samples, the largest concentration of D4 was reported at 32.0 ng·mL^{-1} in primary sludge on March 7, 2014 while the smallest one was 1.4 ng·mL^{-1} in RAS on November 15, 2013. The largest concentration of D5 was reported at 305.7 ng·mL^{-1} in primary digester on January 13, 2014 while the smallest one was 12.3 ng·mL^{-1} in RAS on March 7, 2014.

Table 4-11: Mean Mass Rate of D4 and D5 in Loveland WWTP

	Influent	Primary Sludge	WAS	Effluent
Flow Rate (MGD)	5.4	0.4	0.2	5.4
D4 (g•d^{-1})	47.0	26.6	3.4	45.0
D5 (g•d^{-1})	130.8	109.3	35.1	55.2

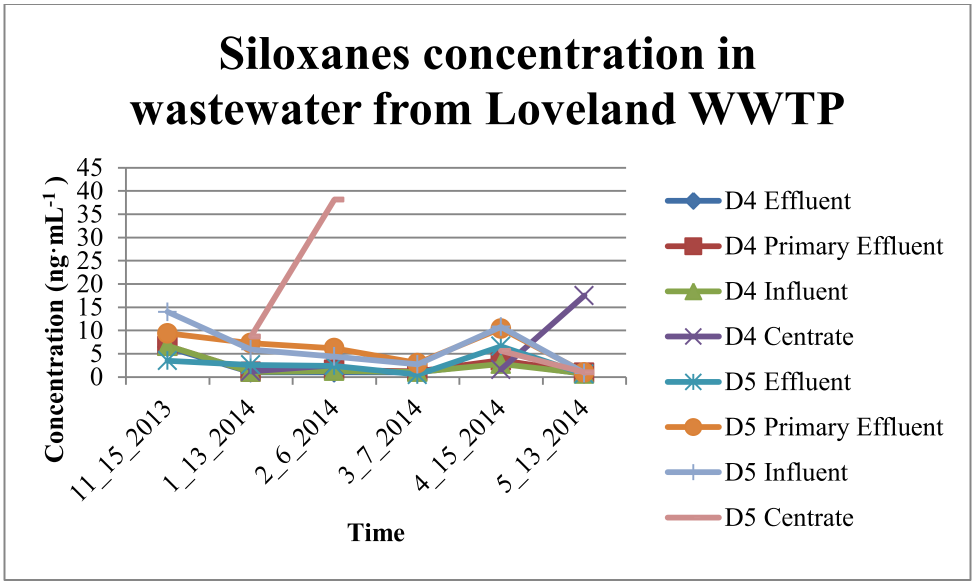


Figure 4-9: Concentrations of D4 and D5 in Wastewater Samples in Loveland WWTP

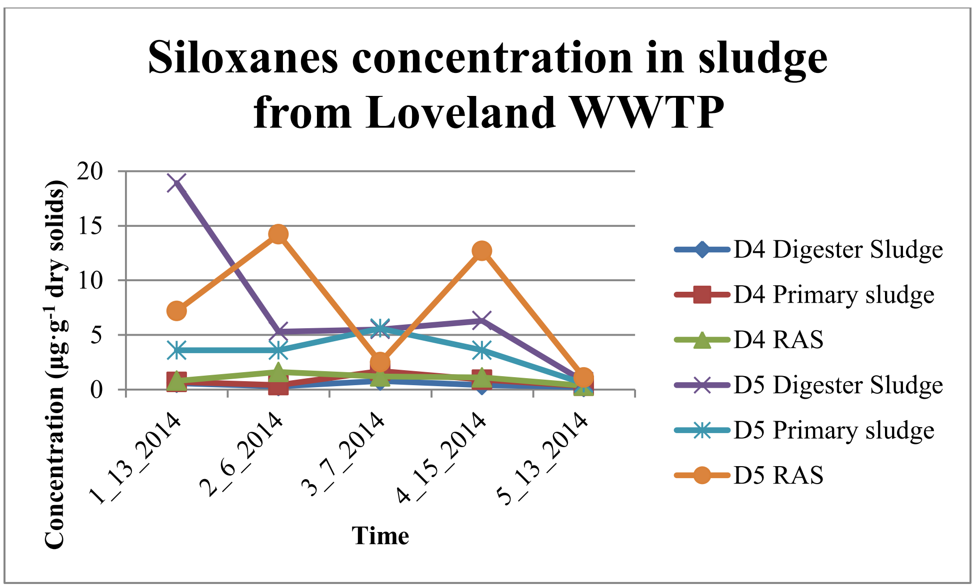


Figure 4-10: Concentrations of D4 and D5 in Sludge Samples in Loveland WWTP

Table 4-12 shows the concentration of D4 and D5 in sludge samples in Loveland WWTP as $\mu g \cdot g^{-1}$ dry solid. Concentration of D4 was significantly less than that of D5 in sludge samples on five testing days. The concentrations of D4 in both digester sludge and primary sludge decreased from January to February and from March to April 2014 while they increased from January to Feburary and from March to April 2014 in return activated sludge. The largest concentration of

D4 was observed at 1.67 $\mu g \cdot g^{-1}$ dry solid in the primary sludge on March 7, 2014 while the smallest was at 0.27 $\mu g \cdot g^{-1}$ dry solid in primary digester on February 6, 2014. Then the largest concentration of D5 was obtained at 18.89 $\mu g \cdot g^{-1}$ dry solid in RAS on January 13, 2014 and the smallest was at 2.52 $\mu g \cdot g^{-1}$ dry solid in primary sludge on March 7, 2014.

Table 4-12: Concentrations of Siloxanes in Sludge Samples in Loveland WWTP

Date			Digester Sludge	Primary Sludge	RAS
1_13_2014	TS ($mg \cdot L^{-1}$)		16180	20820	4150
	Concentration	D4	0.6	0.7	0.8
	($\mu g \cdot g^{-1}$ dry solid)	D5	18.9	3.6	7.2
2_6_2014	TS ($mg \cdot L^{-1}$)		14690	39640	1840
	Concentration	D4	0.3	0.4	1.6
	($\mu g \cdot g^{-1}$ dry solid)	D5	5.3	3.6	14.2
3_7_2014	TS ($mg \cdot L^{-1}$)		15015	19215	4880
	Concentration	D4	0.8	1.7	1.2
	($\mu g \cdot g^{-1}$ dry solid)	D5	5.5	5.6	2.5
4_15_2014	TS ($mg \cdot L^{-1}$)		14715	22500	5100
	Concentration	D4	0.4	0.9	1.1
	($\mu g \cdot g^{-1}$ dry solid)	D5	6.3	3.6	12.7
5_13_2014	TS ($mg \cdot L^{-1}$)		14970	34755	26525
	Concentration	D4	0.2	0.3	0.4
	($\mu g \cdot g^{-1}$ dry solid)	D5	0.7	0.6	1.1

The differences of concentrations on different time were caused by several factors. The weather and temperature not only affect the solubility of siloxanes in water and sludge but also impact the residents habits of using cosmetics and personal care products which are the sources of siloxanes and taking showers. The changes in precipitation events cause different infiltration, which would also change the concentrations of siloxanes in the wastewater.

4.3.6 Concentration Comparison

Tables 4-13 and 4-14 compare the concentrations determined with this study to other studies. This study determined the D4 and D5 concentrations in different samples from the wastewater treatment process. Kulkarni(2012) measured D4 and D5 in Loveland WWTP with the extraction method and reported 0-3.25 $\mu g \cdot g^{-1}$ dry solid of D4 and 1.15-17.11 $\mu g \cdot g^{-1}$ dry solid of D5 in RAS/WAS sample. That study also reported 1 $\mu g \cdot g^{-1}$ dry solid of D5 was present in the RAS/WAS samples in Drake WWRF. Compared to the study done in UK by Egmond et al. (2013), the concentrations in the effluent were higher and the ones in primary sludge were lower in this study. It was suspected in this study that the receiving water body of the wastewater treatment plants would obtain more siloxanes and that the removal amount was inadequate compared to the wastewater treatment in UK analyzed by Egmond et al (2013). The analysis reported in Greece by Bletsou et al. (2013) indicated that the concentrations of D4 were lower and the D5 were about the same level in effluent, influent and dewatered sludge compared to this this study. With different cities, the concentrations of D4 and D5 varied in the local wastewater treatment plants.

The siloxanes in samples from receiving water bodies were below detection level. The average flow rates of Cache La Poudre River (water source of Fossil Creek Ditch) and Big Thompson River are 87 MGD and 47 MGD, which are much larger than the effluent flow rates, so the dispersion and dilution might reduce the concentrations significantly as well as the adsorption onto the river sediment. The predicted concentrations of D4 and D5 were 0.2 $ug \cdot L^{-1}$ and 0.3 $ug \cdot L^{-1}$ each in the Fossil Creak Ditch and Big Thompson River at the discharge points based on the relative river and effluent flowrates and D4 and D5 concentrations in the effluent.

Table 4-13: Concentrations in Wastewater Comparison with Other Studies

Sample ($\mu g \cdot L^{-1}$)	This study USA		van Egmond et al, 2013 UK		Bletsou et al, 2013 Greece		Wang et al, 2012 Canada		Sanchis et al,2012 NE Spain	
	D4	D5	D4	D5	D4	D5	D4	D5	D4	D5
Effluent	0.7-6.4	0.4-6.6	0.024	0.45	0.13	1.8			0.076	0.55
Influent	0.7-11.3	0.9-14.0	<0.2	9.3-10.8	0.15	2.6	0.3-6.7	7.8-135	0.3	8.8
Centrate	0.7-17.5	1.3-38.1								
Primary Effluent	0.96.7	1.0-10.4								

Table 4-14: Concentration in Sludge Comparision with Other Studies

Sample ($\mu g \cdot g^{-1}$ dry solids)	This study Fort Collins and Loveland		van Egmond et al, 2013 UK		Bletsou et al, 2013 Greece		Xu et al, 2013 China		Wang et al, 2012 Canada		Companioni -Damas et al, 2012 NE Spain		Zhang et al, 2011 NE China		Kaj et al, 2005 Sweden	
	D4	D5	D4	D5	D4	D5	D4	D5	D4	D5	D4	D5	D4	D5	D4	D5
Primary Sludge	0.3-1.7	0.6-7.0	<10	46.8-48.4												
TWAS	0.4-1.6	0.6-23.4			0.11	15.1										
RAS/WAS	0.4-1.8	1.0-31.4					0.26-2.3	0.65-3.7	<0.003-0.049	0.011-5.84	2.5-15.1	2.1-82.1	0.1	0.28	0.39	9.5
Digester sludge	0.2-0.8	0.6-21.0	1.53 $ug \cdot L^{-1}$	43.2 $ug \cdot L^{-1}$												

4.4 Conclusions

D4 and D5 were detected using headspace GC/MS method in wastewater and sludge samples collected from two WWTPs in Northern Colorado in this research. D5 was the dominant siloxane, detected at 0.4-38.1 $\mu g \cdot L^{-1}$ in wastewater and 0.6-31.4 $\mu g \cdot g^{-1}$dry solid in the sludge compared to D4 at 0.7-17.5 $\mu g \cdot L^{-1}$ in wastewater and 0.2-1.7 $\mu g \cdot g^{-1}$dry solid in the sludge. The mass loadings of D4 and D5 were tracked in both WWTPs. The effects of the sample matrix on volatilization of siloxanes was observed. Sludge samples had lower levels of siloxanes in the headspace compared to wastewater samples. As expected, the amount of D4 and D5 in effluent was smaller than that in influent, which provides about 29% of D4 and 59% of D5 average removal and lower levels of siloxanes discharged into the receiving water body.

Due to the complex matrix of sludge, different dilution ratios and equipment conditions may be developed to increase the analysis sensitivity. Concentrations of siloxanes in the air at the WWTP could be measured to understand the fate of siloxane in the environment. Because of occurrence of siloxanes, removal treatment is necessary for WWTP operations.

References

Accettola, F., Guebitz, G., Schoeftner, R., (2008). Siloxane removal from biogas by biofiltration: biodegradation studies. Clean Technologies and Environmental Policy, 10, 211-218.

Annelin, R.B., Frye, C.L., (1989). The piscine bioconcentration characteristics of cyclic and linear oligomeric permethylsiloxanes. Science of the Total Environment, 83, 1-11.

Appels, L., Baeyens, J., Dewil R., (2008). Siloxane removal from biosolids by peroxidation. Energy Conversion and Management, 49, 2859-2864.

Atkinson, R. (1991). Kinetics of the gas-phase reactions of a series of organosilicon compounds with OH and NO3 radicals and O3 at 297 ± 2K. Environmental Science and Technology. 25, 863–866.

Bletsou, A.A., Asimakopoulos A.G., Stasinakis, A.S., Thomaidis, N.S., Kannan, K., (2013). Mass Loading and Fate of Linear and Cyclic Siloxanes in a Wastewater Treatment Plant in Greece. Environmental Science and Technology, 47, 1824-1832.

Borga, K., Fjeld, E., Kierkegaard, A., McLachlan, M.S., (2013). Consistency in Trophic Magnification Factors of Cyclic Methyl Siloxanes in Pelagic Freshwater Food Webs Leading to Brown Trout. Environemtal Science and Technology, 47, 14394-14402.

Brooke, D.N., Crookes, M.J., Gray, D., Robertson, S., (2009). Environmental Risk Assessment Report: Decamethylcyclopentasiloxane. Environment Agency of England and Wales, Bristol

Bruggeman, W.A., Weber-Fung, D., Opperhuizen, A., van der Steen, J., Wijbenga, A., Hutzinger, O., 1984. Absorption and retention of polydimethylsiloxanes (silicones) in fish: preliminary experiments. Toxicol. Environ. Chem. 7, 287–296.

City of Loveland. (2013). Wastewater. Retrieved from City of Loveland: http://www.cityofloveland.org/index.aspx?page=859

Companioni-Damas, E.Y., Santos, F.J., Galceran, M.T., (2011). Analysis of linear and cyclic methylsiloxanes in water by headspace-solid phase microextraction and gas chromatography–mass spectrometry. Talanta, 89, 63-69.

Companioni-Damas, E.Y., Santos, F.J., Galceran, M.T., (2012). Analysis of linear and cyclic methylsiloxanes in sewage sludges and urban soils by concurrent solvent recondensation – large volume injection – gas chromatography–mass spectrometry. Journal of Chromatography A, 1268, 150-156.

Dewil, R., Appels, L., Baeyens, J., (2006). Energy use of biogas hampered by the presence of siloxanes. Energy Conversion & Management, 47, 1711-1722.

Dewil, R., Appels, L., Baeyens, J., Buczynska, A., Van Vaeck, L., (2007). The analysis of volatile siloxanes in waste activated sludge. Talanta, 74, 14-19.

Drottar, K.R., (2005a). 14C-Decamethylcyclopentasiloxane (14C-D5): Bioconcentration in the Fathead Minnow (Pimphales promelas) under Flow-Through Test Conditions. Dow Corning Corporation, Silicones Environment, Health and Safety Council (SEHSC).

Environment Canada and Health Canada. (2008). Screening Assessment for the Challenge Octamethylcyclotetrasiloxane. Retrieved from: http://www.ec.gc.ca/substances/ese/eng/challenge/batch2/batch2_541-02-6_en.pdf.

European Commission. (2010). Amending Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) as Regards Annex XIII. Retrieved from: <http://register.consilium.europa.eu/pdf/en/10/st14/st14860.en10.pdf>.

Fackler, P.H., Dionne, E., Hartley, D.A., Hamelink, J.L., (1995). Bioconcentration by fish of a highly volatile silicone compound in a totally enclosed aquatic exposure system. Environmental Toxicology and Chemistry, 14, 1649-1656.

Giesy, J.P., Solomon, K., Kacew, S., et al. (2011). Report of the Board of Review for Decamethylcyclopentasiloxane (D5). Siloxane D5 Board of Review, Ottawa, ON, Canada. p. 83.

Gouin, T., van Egmond, R., Sparham, C., Hastie, C., Chowdhury, N., (2013). Simulated use and wash-off release of decamethylcyclopentasiloxane used in anti-perspirants. Chemosphere, 93(5), 726-734.

Griffin P., Severn Trent Water Wastewater Treatment Plant at Minworth. In: ST-seminar 2004.

Hagmann, M., Heimbrand, E., Hentschel, P., (1999). Determination of siloxanes in biogas from landfills and sewage treatment plants. Seventh international waste management and landfill symposium. Cagliari, Italy.

Hagmann, M., Hesse, E., Hentschel, P., Bauer, T., (2001). Purification of biogas-removal of volatile silicones. Eighth international waste management and landfill symposium, II(641-644). Sardinia.

Hayes H., Graening, G.J., Saeed, S., Kao, S., (2003). A Summary of Available Analytical Methods for the Determination of Siloxanes in Biogas. Retrieved from Airtoxics: http://www.airtoxics.com/literature/Siloxanes_SWANA_03.pdf

Huber, L. (2010). Validation of Analytical Methods. Germany: Agilent Technologies.

Hunter, M.J., Hyde, J.F., Warrick, E.L., Fletcher, H.J., (1946). Organo-Silicon Polymers: The Cyclic Dimthyl Siloxanes. Journal of the American Chemical Society, 68, 667-672.

Kaj, L., Andersson, J., Palm Cousins, A., Schmidbauer, N., Brorstrom-Lunden, E., Cato, I., (2004). Results from the Swedish National Screening Programme. Swedish Environmental Research Institute Ltd.

Kaj, L., Andersson, J., Palm Cousins, A., Schmidbauer, N., Brorstrom-Lunden, E., Cato, I., (2005a). Results from the Swedish National Screening Programme 2004. Subreport 4: Siloxanes. Stockholm: IVL Swedish Environmental Research Institute Ltd.,.

Kaj, L., Schlabach, M., Andersson, J., Palm Cousins, A., Schmidbauer, N., Brorstrom-Lunden, E., (2005b). Siloxanes in the Nordic Environment. Nordic Council of Ministers, Copenhagen, TemaNord.

Kent, D.J., McNamara, P.C., Putt, A.E., Hobson, J.F., Silberhorn, E.M., 1994. Octamethylcyclotetrasiloxane in aquatic sediments: toxicity and risk assessment. Ecotoxicology and Environmental Safety, 29, 372–389.

Kierkegaard, A., Bignert, A., McLachlan, M.S., (2012). Cyclic volatile methylsiloxanes in fish from the Baltic Sea. Chemosphere, 93(5), 774-778.

Kierkegaard, A., Bignert, A., McLachlan, M.S., (2013). Bioaccumulation of Decamethylcyclopentasiloxane in Perch in Swedish Lake. Chemosphere, 93, 789-793.

Kozerski, G., 2007. Determination of the 1-Octanol/Water Partition Coefficient of Decamethylcyclopentasiloxane (D5) by the Slow-Stirring Method Using Gas Chromatography and Mass Spectrometry. Silicones Environmental, Health, and Safety Council (SEHSC) (cited from the Report of the Assessment for D5 by Environment Canada and Health Canada).

Krueger, H.O., Thomas, S.T., Kendall, T.Z., (2008a). D4: A Prolonged Sediment Toxicity Test with Chironomus riparius Using Spiked Sediment. Canada: Wildlife International, LTD.

Krueger, H.O., Thomas, S.T., Kendall, T.Z., (2008b). D5: A Prolonged Sediment Toxicity Test with Chironomus riparius Using Spiked Sediment. Canada: Wildlife International, LTD.

Kulkarni, H. V., (2012). Occurrence of Cyclo-Siloxanes in Wastewater Treatment Plant-Quantification and Monitoring. Master Thesis. Colorado State Univeristy.

Lee S., Cho W., Song T. (2001). Removal process for octamethylcyclotetrasiloxane from biogas in sewage treatment plant. Journal of Industrial and Engineering Chemistry, 28, 276-280.

Looney, R.J., Frampton, M.W., Byam, J., Kenaga, C., Speers, D.M., Cox., C, Mast, R., Klykken, P.C., Morrow, P.E., Utell, M.J., (1998). Acute Respiratory Exposure of Human

Volunteers to Octamethylcuclotetrasiloxane (D4): Absence of Immunological Effects. Toxicological Science, 44, 214-220.

McBean, E.A. (2008a). Siloxanes in biogases from landfills and wastewater digesters. Canadian Journal of Civil Engineering, 35, 431-436.

McBean, E. A-(2008b). NRC Research Press. Retrieved from NRC Research Press: http://www.nrcresearchpress.com/doi/pdf/10.1139/L07-144

Miller, J., 2007. Soil–Water Distribution of Octamethylcyclotetrasiloxane (D4) Using a Batch Equilibrium Method. Draft Report. Centre Europeen des Silicones (CES). Cited from the Report of the Assessment for D4 by Environment Canada and Health Canada.

Montemayor et al. (2013). Evaporative fate of cyclopentasiloxane (D5) from personal care products during product use: antiperspirants, skin care products and hair care products. Chemosphere, 93(5), 711-725.

Mueller, J.A, Di Toro, D.M., Maiello J.A., (1995). Fate of octamethylcyclotetrasiloxane (OMCTS) in the atmosphere and in sewage treatment plants as an estimation of aquatic exposure. Environ Toxicol Chem, 14, 1657-1666.

Opperhuizen, A., Damen, H.W.J., Asyee, G.M., Van Der Steen, J.M.D., Hutzinger, O., (1987). Uptake and elimination by fish of polydimethylsiloxanes (silicones) after dietary and aqueous exposure. Toxicological and Environmental Chemistry, 13, 265-285.

Parrott, J., Alaee, M., Wang, D., Sverko, E., (2013). Fathead minnow (Pimephales promelas) egg-to-juvenile exposure to decamethylcyclopentasiloxane (D5). Chemosphere, 93, 813-818

Pellizzari, E.D., Bunch, J.E., Berkley, R.E., McRae, J., (1976). Determination of trace hazardous organic vapor pollutants in ambient atmospheres by gas chromatography/mass spectrometry/computer. Analytical Chemistry, 48, 803-807.

Popat S., Deshusses M. (2008). Biological removal of siloxanes from landfill and digester gases: opportunitues and challenges. Environmental Science and Technology, 42, 8510-8515.

Rasi, Lehtinen, J., Rintala, J., (2010). Determination of organic silicon compounds in biogas from wastewater treatments plants, landfills, and co-digestion plants. Revewable Energy, 35, 2666-2673.

Rossol D., Schmelz K.G., Hohmann R. (2003). Siloxane im Faulgas. KA-AbwasserAbfall, 8, 8.

Sanchis, J., Martinez, E., Ginebreda, A., Farre, M., Barcelo, D., (2013). Occurrence of linear and cyclic volatile methylsiloxanes in wastewater, surface water and sediments from Catalonia. Science of the Total Environment, 443, 530-538.

Sanchez-Brunete C., Miguel, E., Albero, B., Tadeo, J.L., (2010). Determination of cyclic and linear siloxanes in soil samples by ultrasonic-assisted extraction and gas chromatography–mass spectrometry. Journal of Chromatography A, 1217, 7024-7030.

Schweighkofler, M., Niessner, R., (2001). Removal of siloxanes in biogases. Journal of Hazardous Materials, 83, 183-196.

Shields H.C., Fleischer, D.M., Weschler, C.J., (1996). Comparisons among VOCs measured in three types of US commercial buildings with different occupant densities. Indoor Air, 6, 2-7.

Smith, J.A., Witkowski, P.J., Fusillo, T.V., 1988, Manmade organic compounds in the surface waters of the United States--A review of current understanding: U.S. Geological Survey Circular 1007, 92.

Sousa J., McNamara, P.C., Putt, A.E., Machado, M.W., Surprenant, D.C., Hamelink, J.L., Kent, D.J., Silberhorn, E.M., Hobson, J.F., (1995). Effects of octamethylcyclotetrasiloxane (OMCTS) on freshwater and marine organisms. Environmental toxicology and chemistry, 14, 1639-1647.

Sparham C., Van Egmond, R., O'Connor, S., Hastie, C., Whelan, M., Kanda, R., Franklin, O., (2008). Determination of decamethylcyclopentasiloxane in river water and final effluent by headspace gas chromatography/mass spectrometry. Journal of Chromatography A, 1212, 124-129.

Steer A, M., Smyth, S.A., (2008). Measurement of volatile methylsiloxanes in air by thermal desorption GC/MS. In: Alaee, M., Steer, H. (Eds.), First Annual Workshop on Organosilicon Compounds in the Environment. Aquatic Ecosystem Protection Research Division, Water Science and Technology Directorate, Environment Canada, Burlington, Canada.

United Nations Office on Drugs and Crime. (2009). Guidance for the Validation of Analytical Methodology and Calibration of Equipment used for Testing of Illicit Drugs in Seized Materials and Biological Specimens. Austria: United Nations.

US EPA. (2009). Siloxane D5 in Drycleaning Applications Fact Sheet. Retrieved from EPA: http://www.epa.gov/oppt/dfe/pubs/garment/d5fs3.pdf

US EPA. (2011). Opportunities for Combined Heat and Power at Wastewater Treatment Facilities: Market Analysis and Lessons from the Field. US EPA.

van Egmond, R., Sanders, D., 2010. Investigations into the effect of ageing on the bioavailability of decamethylcyclopentasiloxane in artificial and natural sediments. In: Poster presentation at SETAC Europe 20th Annual Meeting, Seville, 23–27 May 2010.

Varaprath, S., Frye, C.L., Hamelink, J., (1996). Aqueous solubility of permethylsiloxanes (silicones). Environmental Toxicology and Chemistry, 15, 1263-1265.

Wang, D.-G., Norwood, W., Alaee, M., Byer, J.D., Brimble, S., (2012). Review of recent advances in research on the toxicity, detection, occurrence and fate of cyclic volatile. Chemosphere.

Wang D.-G., Steer, H., Tait, T., Williams, Z., Pacepavicius, G., Young, T., Ng, T., Smyth, S.A., Kinsman, L., Alaee, M., (2013). Concentrations of Cyclic Volatile Methylsiloxanes in Biosolid Amended Soil, Influent, Effluent, Receiving Water, and Sediment of Wastewater Treatment Plants in Canada. Chemosphere, 93(5), 766-773.

Wang, X.M., Lee, S.C., Sheng, G.Y., Chan, L.Y., Fu, J.M., Li, X.D., Min, Y.S., Chan, C.Y., (2001). Cyclic organosilicon compounds in ambient air in Guangzhou, Macau and Nanhai, Pearl River Delta. Applied Geochemistry, 16, 1447-1454.

Warner N.A., Evenset, A., Christensen, G., Gabrielsen, G.W., Borga, K., Leknes, H., (2010). Volatile siloxanes in the European arctic: assessment of sources and spatial distribution. Environmental Science and Technology, 44, 7705-7710.

Water Environment Research Fundation. (2012). Barriers to Biogas Use for Renewable Energy. Retrieved from Werf.org: https://www.werf.org/a/ka/Search/ResearchProfile.aspx?ReportId=OWSO11C10

Wheless E. Pierce, J., (2004). Siloxanes in Landfill and Digester Gas Update. 27th SWANA Landfill Gas symposium, (pp. 22-25). San Antonio, Texas.

Xu H., Zhang, X., Jin, F., Hua, D., Zhang, J., Li, Y., (2012). Progress on Technology of Siloxane Removal from Biogas. China Biogas, 30(6), 18-22.

Yucuis, R.A., Stanier, C.O., Hornbuchle, K.C., (2013). Cyclic siloxanes in Air, including Identification of High Levels in Chicago and Distinct Diurnal Variation. Chemoshoere, 92, 905-910.

Zhang P., Zhang G., Wang W. (2007). Ultrasonic treatment of biological sludge: Floc disintegration, cell analysis and inactivation. Bioresource Technology, 98(1), 207-210.

Zhang, Z., Qi, H., Ren, N., Li, Y., Gao, D., Kannan, K., (2011). Survey of cyclic and linear siloxanes in sediment from the Songhua River and in sewage sludge from wastewater treatment plants, Northeastern China. Archives of Environmental Contamination and Toxicology, 60, 204-211.

Appendix

A. Raw Data – D4

Table A-1: Data of D4 during feasibility test on 4/4/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
4_4_13 D4 D5 Standard	Analyte	50	5.12	1375848.9
4_4_13 Sludge	Analyte		5.13	7017.1
4_4_13 Sludge + 3ug	Analyte		5.13	168616.2
4_4_13 Sludge + 10ug	Analyte		5.13	698807.8
4_4_13 3ug std	Standard	150	5.13	4228780.0
4_4_13 10ug std	Standard	500	5.12	6703429.5

Table A-2: Data of D4 analysis on 5/7/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
05-07-13_siloxane_0	Standard	0	5.13	486.0
05-07-13_siloxane_25ngml	Standard	25	5.13	4941.8
05-07-13_siloxane_100ngml	Standard	100	5.13	2563.7
05-07-13_siloxane_250ngml	Standard	250	5.13	64745.8
05-07-13_siloxane_0_sludge1	Analyte		5.13	964.6
05-07-13_siloxane_0_sludge2	Analyte		5.13	2030.8
05-07-13siloxane_25_sludge1	Analyte	25	5.13	293.1
05-07-13siloxane_25_sludge2	Analyte	25	5.13	2144.5
05-07-13siloxane_50_sludge1	Analyte	50	5.13	12930.1
05-07-13siloxane_50_sludge2	Analyte	50	5.13	9916.9
05-07-13siloxane_75_sludge1	Analyte	75	5.12	16194.1
05-07-13siloxane_75_sludge2	Analyte	75	5.13	38118.4
05-07-13siloxane_100_sludge1	Analyte	100	5.13	539.9
05-07-13siloxane_100_sludge2	Analyte	100	5.13	675.9
05-07-13siloxane_250_sludge1	Analyte	250	5.13	3705.5
05-07-13siloxane_250_sludge2	Analyte	250	5.13	3265.6

Table A-3: Data of D4 analysis on 7/12/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
7_12_13_Siloxane_Sample 1	Analyte		5.17	38.4

7_12_13_Siloxane_Sample 2	Analyte		5.1	417.0
7_12_13_Siloxane_0	Blank		4.9	780.5
7_12_13_Siloxane_50ngmL	Standard	50	5.02	59.6
7_12_13_Siloxane_100ngmL	Standard	100	4.72	95.4
7_12_13_Siloxane_500ngmL	Standard	500	5.27	194.4
7_12_13_Siloxane_Sample 3	Analyte		5.17	38.4

Table A-4: Data of D4 analysis for Fort Collins and Loveland on 7/18/13

Name	Type	Std. Conc ($ng\cdot mL^{-1}$)	RT	Area
7_18_13_Siloxane_Blank1	Blank		1.89	201.2
7_18_13_Siloxane_Post1	Analyte		1.9	1506.1
7_18_13_Siloxane_Post2	Analyte		1.9	1228.5
7_18_13_Siloxane_Pre1	Analyte		1.9	1467.4
7_18_13_Siloxane_Pre2	Analyte		1.9	1627.7
7_18_13_Siloxane_LL1	Analyte		1.9	1796.4
7_18_13_Siloxane_LL2	Analyte		1.9	2017.3
7_18_13_Siloxane_Blank2	Blank	0	1.9	90.0
7_18_13_Siloxane_5ngmL	Standard	5	1.89	3614.7
7_18_13_Siloxane_10ngmL	Standard	10	1.89	6578.9
7_18_13_Siloxane_25ngml	Standard	25	1.89	18128.3
7_18_13_Siloxane_Blank3	Blank	0	1.89	122.2

Table A-5: Data of D4 analysis on 9/4/13

Name	Type	Std. Conc ($ng\cdot mL^{-1}$)	RT	Area
9_4_13 Siloxane_Blank1	Blank		5.01	5086.6
9_4_13 Siloxane_1ngml	Standard	1	5.01	5228.3
9_4_13 Siloxane_2_5ngml	Standard	2.5	5.01	4449.4
9_4_13 Siloxane_7_5ngml	Standard	7.5	5.01	3865.7
9_4_13 Siloxane_Blank2	Blank		5.01	4740.3
9_4_13 Siloxane_sludge1	Analyte		5.01	1470.5
9_4_13 Siloxane_sludge2	Analyte		5.01	1237.3
9_4_13 Siloxane_Blank3	Blank		5.01	2242.2

Table A-6: Data of D4 analysis on 9/10/13

Name	Type	Std. Conc ($ng\cdot mL^{-1}$)	RT	Area
9_10_13 Siloxane	Blank		4.97	424.9

9_10_13 Siloxane_blank1	Blank		4.99	13418.7
9_10_13 Siloxane1ngml	Standard	1	4.99	3397.3
9_10_13 Siloxane2_5ngml	Standard	2.5	4.99	3111.9

Table A-7: Data of D4 during blank test on 9/11/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_11_2013 Acetone	Blank		4.96	322.7
9_11_2013 Water	Blank		4.99	673.5
9_11_2013 IS	Analyte		4.99	560.9

Table A-8: Data of D4 analysis repeated on 9/11/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_12_2013 Siloxaneblank	Blank	0	4.98	205.4
9_12_2013 Siloxane1ngml	Standard	1	4.98	240.3
9_12_2013 Siloxane2_5ngml	Standard	2.5	4.98	220.5
9_12_2013 Siloxane7_5ngml	Standard	7.5	4.99	217.1

Table A-9: Data of D4 analysis on 9/12/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_12_2013 Siloxaneblank	Blank	0	4.98	205.4
9_12_2013 Siloxane1ngml	Standard	1	4.98	240.3
9_12_2013 Siloxane2_5ngml	Standard	2.5	4.98	220.5
9_12_2013 Siloxane7_5ngml	Standard	7.5	4.99	217.1

Table A-10: Data of D4 in water analysis on 9/30/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
2013_9_30Siloxans_water 1	Standard	10	2.01	441974.6
2013_9_30Siloxans_water 2	Standard	50	2.01	1660765.1
2013_9_30Siloxans_water 3	Standard	100	2.01	4001189.8

Table A-11: Data of D4 in sludge analysis on 9/30/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
2013_9_30Siloxans_sludge 1	Blank	0	2.03	9847.3
2013_9_30Siloxans_sludge 2	Standard	10	2.03	199841.4
2013_9_30Siloxans_sludge 3	Standard	50	2.03	1040322.5
2013_9_30Siloxans_sludge 4	Standard	100	2.03	2313130.5

Table A-12: Data of D4 analysis on 10/1/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
2013_10_1 Siloxane 0ngmL	Blank	0	2.02	5698.0
2013_10_1 Siloxane 10ngmL	Analyte	10	2.02	197124.7

Table A-13: Data of D4 analysis with initial temperature of 60 ℃ on 10/4/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_4_2013 Siloxanes IS 60 C	Blank	25	2.57	303428.3

Table A-14: Data of D4 analysis with initial temperature of 70 ℃ on 10/4/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_4_2013 Siloxane full scan	Analyte	25		
10_4_2013 Siloxane IS with 70C	Analyte	50	2.35	1119280.3

Table A-15: Data of D4 analysis with initial temperature of 80 ℃ on 10/7/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_7_13 Siloxane at 80C	Analyte	50	2.2	750802.8

Table A-16: Data of D4 analysis with initial temperature of 75 ℃ on 10/7/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_7_13 Siloxane at 75C		50	2.31	748411.0

Table A-17: Data of D4 QAQC analysis with split ratio of 1: 20 on 10/8/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area

10_8_13 slixane QAQC Blank1	Blank	0	2.31	789.5
10_8_13 slixane QAQC 10ngmLwater1	Standard	10	2.32	247547.7
10_8_13 slixane QAQC 100ngmL water1	Standard	100	2.32	2631686.5
10_8_13 slixane QAQC 1000ngmLwater1	Standard	1000	2.31	12631592.0
10_8_13 slixane QAQC 10ngmLwater2	Standard	10	2.31	296764.4
10_8_13 slixane QAQC 100ngmLwater2	Standard	100	2.32	1943104.1
10_8_13 slixane QAQC 1000ngmLwater2	Standard	1000	2.32	12535840.0
10_8_13 slixane QAQC 10ngmLwater3	Standard	10	2.32	259104.3
10_8_13 slixane QAQC 100ngmLwater3	Standard	100	2.32	2349425.8
10_8_13 slixane QAQC 1000ngmLwater3	Standard	1000	2.32	12510815.0
10_8_13 slixane QAQC Blank2 sludge	Blank	0	2.32	7187.1
10_8_13 slixane QAQC 10ngmL sludge1	Analyte	10	2.32	160356.4
10_8_13 slixane QAQC 100ngmL sludge1	Analyte	100	2.32	1364234.4
10_8_13 slixane QAQC 1000ngmL sludge1	Analyte	1000	2.32	9437333.0
10_8_13 slixane QAQC Blank3 sludge	Blank	0	2.32	5679.6
10_8_13 slixane QAQC 10ngmL sludge2	Analyte	10	2.32	128460.7
10_8_13 slixane QAQC 100ngmL sludge2	Analyte	100	2.32	983308.1
10_8_13 slixane QAQC 1000ngmL sludge2	Analyte	1000	2.32	9240279.0
10_8_13 slixane QAQC Blank4 sludge	Blank	0	2.32	5260.3
10_8_13 slixane QAQC 10ngmL sludge3	Analyte	10	2.32	111489.4
10_8_13 slixane QAQC 100ngmL sludge3	Analyte	100	2.32	941857.8
10_8_13 slixane QAQC 1000ngmL sludge3	Analyte	1000	2.32	8904177.0

Table A-18: Data of D4 QAQC analysis with split ratio of 1: 20 on 10/9/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_9_13 slixane QAQC Blank1	Blank	0	2.31	568.0
10_9_13 slixane QAQC 10ngmLwater1	Standard	10	2.31	88494.0
10_9_13 slixane QAQC 100ngmL water1	Standard	100	2.31	1161494.0
10_9_13 slixane QAQC 1000ngmLwater1	Standard	1000	2.31	8283428.0
10_9_13 slixane QAQC 10ngmLwater2	Standard	10	2.31	86984.0
10_9_13 slixane QAQC 100ngmLwater2	Standard	100	2.31	898026.0
10_9_13 slixane QAQC 1000ngmLwater2	Standard	1000	2.32	7234281.5
10_9_13 slixane QAQC 10ngmLwater3	Standard	10	2.31	90139.9
10_9_13 slixane QAQC 100ngmLwater3	Standard	100	2.31	730478.0
10_9_13 slixane QAQC 1000ngmLwater3	Standard	1000	2.31	7210900.5
10_9_13 slixane QAQC 5ngmLwater	Analyte	5	2.31	37215.4
10_9_13 slixane QAQC 25ngmLwater	Analyte	25	2.31	188057.4
10_9_13 slixane QAQC 500ngmLwater	Analyte	500	2.32	4110015.0

10_9_13 slixane QAQC Blank2 sludge	Blank	0	2.32	1639.0
10_9_13 slixane QAQC 10ngmL sludge1	Analyte	10	2.32	41840.9
10_9_13 slixane QAQC 100ngmL sludge1	Analyte	100	2.32	628190.4
10_9_13 slixane QAQC 1000ngmL sludge1	Analyte	1000	2.32	4484624.5
10_9_13 slixane QAQC Blank3 sludge	Blank	0	2.32	1417.3
10_9_13 slixane QAQC 10ngmL sludge2	Analyte	10	2.32	37299.9
10_9_13 slixane QAQC 100ngmL sludge2	Analyte	100	2.32	395702.6
10_9_13 slixane QAQC 1000ngmL sludge2	Analyte	1000	2.32	4131980.3
10_9_13 slixane QAQC Blank4 sludge	Blank	0	2.32	1297.8
10_9_13 slixane QAQC 10ngmL sludge3	Analyte	10	2.32	38050.4
10_9_13 slixane QAQC 100ngmL sludge3	Analyte	100	2.32	442591.7
10_9_13 slixane QAQC 1000ngmL sludge3	Analyte	1000	2.32	5759707.0
10_9_13 slixane QAQC 5ngmL sludge	Analyte	5	2.32	27147.2
10_9_13 slixane QAQC 25ngmL sludge	Analyte	25	2.32	121449.1
10_9_13 slixane QAQC 500ngmL sludge	Analyte	500	2.32	2384248.0

Table A-19: Data of D4 QAQC analysis with split ratio of 1: 20 on 10/10/13

Name	Type	Std. Conc ($ng{\cdot}mL^{-1}$)	RT	Area
10_10_13 slixane QAQC Blank1	Blank	0	2.32	657.2
10_10_13 slixane QAQC 10ngmLwater1	Standard	10	2.32	59564.7
10_10_13 slixane QAQC 100ngmL water1	Standard	100	2.32	781871.2
10_10_13 slixane QAQC 1000ngmLwater1	Standard	1000	2.32	4093726.5
10_10_13 slixane QAQC 10ngmLwater2	Standard	10	2.32	70724.5
10_10_13 slixane QAQC 100ngmLwater2	Standard	100	2.32	451353.7
10_10_13 slixane QAQC 1000ngmLwater2	Standard	1000	2.32	4423259.5
10_10_13 slixane QAQC 10ngmLwater3	Standard	10	2.32	48393.6
10_10_13 slixane QAQC 100ngmLwater3	Standard	100	2.33	387738.2
10_10_13 slixane QAQC 1000ngmLwater3	Standard	1000	2.32	6355392.0
10_10_13 slixane QAQC 5ngmLwater	Analyte	5	2.32	32585.9
10_10_13 slixane QAQC 25ngmLwater	Analyte	25	2.32	123019.6
10_10_13 slixane QAQC 500ngmLwater	Analyte	500	2.33	2617263.3
10_10_13 slixane QAQC Blank2 sludge	Blank	0	2.33	1374.9
10_10_13 slixane QAQC 10ngmL sludge1	Standard	10	2.33	33610.3
10_10_13 slixane QAQC 100ngmL sludge1	Standard	100	2.33	372099.5
10_10_13 slixane QAQC 1000ngmL sludge1	Standard	1000	2.33	2636830.0
10_10_13 slixane QAQC Blank3 sludge	Blank	0	2.33	1237.9
10_10_13 slixane QAQC 10ngmL sludge2	Standard	10	2.33	26648.3
10_10_13 slixane QAQC 100ngmL sludge2	Standard	100	2.33	393120.4

10_10_13 slixane QAQC 1000ngmL sludge2	Standard	1000	2.33	3245422.0
10_10_13 slixane QAQC Blank4 sludge	Blank	0	2.33	3906.2
10_10_13 slixane QAQC 10ngmL sludge3	Standard	10	2.33	31193.8
10_10_13 slixane QAQC 100ngmL sludge3	Standard	100	2.33	171476.6
10_10_13 slixane QAQC 1000ngmL sludge3	Standard	1000	2.33	4212785.0
10_10_13 slixane QAQC 5ngmL sludge	Analyte	5	2.33	24605.1
10_10_13 slixane QAQC 25ngmL sludge	Analyte	25	2.33	106390.4
10_10_13 slixane QAQC 500ngmL sludge	Analyte	500	2.33	1480711.4

Table A-20: Data of D4 during feasible test of split ratio on 11/1/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
11_1_2013 siloxanes sludge 0ngmL 1:5 split	Analyte		2.49	176617.5
11_1_2013 siloxanes water 50ngmL 1:5 split	Analyte	50	2.5	87.7
11_1_2013 siloxanes sludge 0ngmL 1:10 split	Analyte		2.37	578629.2
11_1_2013 siloxanes water 50ngmL 1:10 split	Analyte	50	2.38	2377.7

Table A-21: Data of D4 QAQC analysis with split ratio 1:10 on 11/2/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
11_2_2013 siloxanes water blank1	Blank		2.38	4998.6
11_2_2013 siloxanes water 01ngmL 1	Standard	0.1	2.38	6827.9
11_2_2013 siloxanes water 05ngmL 1	Standard	0.5	2.38	7711.4
11_2_2013 siloxanes water 1 ngmL 1	Standard	1	2.38	12002.3
11_2_2013 siloxanes water 5 ngmL 1	Standard	5	2.38	40651.1
11_2_2013 siloxanes water 10 ngmL 1	Standard	10	2.38	54679.1
11_2_2013 siloxanes water 50 ngmL 1	Standard	50	2.38	480199.1
11_2_2013 siloxanes water blank 2	Blank		2.38	3925.7
11_2_2013 siloxanes water 01ngmL 2	Standard	0.1	2.38	5982.3
11_2_2013 siloxanes water 05ngmL 2	Standard	0.5	2.38	6715.1
11_2_2013 siloxanes water 1 ngmL 2	Standard	1	2.38	9987.9
11_2_2013 siloxanes water 5 ngmL 2	Standard	5	2.38	42647.4
11_2_2013 siloxanes water 10 ngmL 2	Standard	10	2.38	63553.7
11_2_2013 siloxanes water 50 ngmL 2	Standard	50	2.38	386700.2
11_2_2013 siloxanes water blank 3	Blank		2.38	3949.8
11_2_2013 siloxanes water 01ngmL 3	Standard	0.1	2.38	4408.8
11_2_2013 siloxanes water 05ngmL 3	Standard	0.5	2.38	6726.1
11_2_2013 siloxanes water 1 ngmL 3	Standard	1	2.38	7934.2
11_2_2013 siloxanes water 5 ngmL 3	Standard	5	2.38	24685.2

11_2_2013 siloxanes water 10 ngmL 3	Standard	10	2.38	56378.1
11_2_2013 siloxanes water 50 ngmL 3	Standard	50	2.38	332541.6
11_2_2013 siloxanes sludge blank1	Blank		2.38	2697.6
11_2_2013 siloxanes sludge 01ngmL 1	Analyte		2.38	2772.3
11_2_2013 siloxanes sludge 05ngmL 1	Analyte		2.38	3359.9
11_2_2013 siloxanes sludge 1 ngmL 1	Analyte		2.38	4084.0
11_2_2013 siloxanes sludge 5 ngmL 1	Analyte		2.38	14111.9
11_2_2013 siloxanes sludge 10 ngmL 1	Analyte		2.38	19852.4
11_2_2013 siloxanes sludge 50 ngmL 1	Analyte		2.38	124466.1
11_2_2013 siloxanes sludge blank 2	Blank		2.38	2672.0
11_2_2013 siloxanes sludge 01ngmL 2	Analyte		2.38	3025.8
11_2_2013 siloxanes sludge 05ngmL 2	Analyte		2.38	3429.0
11_2_2013 siloxanes sludge 1 ngmL 2	Analyte		2.38	4048.1
11_2_2013 siloxanes sludge 5 ngmL 2	Analyte		2.38	14268.4
11_2_2013 siloxanes sludge 10 ngmL 2	Analyte		2.38	20350.7
11_2_2013 siloxanes sludge 50 ngmL 2	Analyte		2.38	143275.7
11_2_2013 siloxanes sludge blank 3	Blank		2.38	2378.4
11_2_2013 siloxanes sludge 01ngmL 3	Analyte		2.38	3150.9
11_2_2013 siloxanes sludge 05ngmL 3	Analyte		2.38	3305.8
11_2_2013 siloxanes sludge 1 ngmL 3	Analyte		2.38	4272.1
11_2_2013 siloxanes sludge 5 ngmL 3	Analyte		2.38	11745.3
11_2_2013 siloxanes sludge 10 ngmL 3	Analyte		2.38	19402.1
11_2_2013 siloxanes sludge 50 ngmL 3	Analyte		2.38	120799.9

Table A-22: Data of D4 QAQC analysis with split ratio 1:10 on 11/3/13

Name	Type	Std. Conc ($ng\cdot mL^{-1}$)	RT	Area
11_3_2013 siloxanes water blank1	Blank		2.38	87.0
11_3_2013 siloxanes water 01ngmL 1	Standard	0.1	2.38	1403.7
11_3_2013 siloxanes water 05ngmL 1	Standard	0.5	2.38	3109.6
11_3_2013 siloxanes water 1 ngmL 1	Standard	1	2.38	5638.4
11_3_2013 siloxanes water 5 ngmL 1	Standard	5	2.39	27867.0
11_3_2013 siloxanes water 10 ngmL 1	Standard	10	2.38	57773.8
11_3_2013 siloxanes water 50 ngmL 1	Standard	50	2.39	347507.3
11_3_2013 siloxanes water blank 2	Blank		2.38	235.4
11_3_2013 siloxanes water 01ngmL 2	Standard	0.1	2.38	1259.0
11_3_2013 siloxanes water 05ngmL 2	Standard	0.5	2.39	2685.1
11_3_2013 siloxanes water 1 ngmL 2	Standard	1	2.38	4637.6
11_3_2013 siloxanes water 5 ngmL 2	Standard	5	2.39	26967.9

11_3_2013 siloxanes water 10 ngmL 2	Standard	10	2.39	42476.5
11_3_2013 siloxanes water 50 ngmL 2	Standard	50	2.39	307338.0
11_3_2013 siloxanes water blank 3	Blank		2.38	200.9
11_3_2013 siloxanes water 01ngmL 3	Standard	0.1	2.39	983.9
11_3_2013 siloxanes water 05ngmL 3	Standard	0.5	2.39	2154.1
11_3_2013 siloxanes water 1 ngmL 3	Standard	1	2.39	3540.9
11_3_2013 siloxanes water 5 ngmL 3	Standard	5	2.39	20985.7
11_3_2013 siloxanes water 10 ngmL 3	Standard	10	2.39	38704.7
11_3_2013 siloxanes water 50 ngmL 3	Standard	50	2.38	304592.2
11_3_2013 siloxanes sludge blank1	Blank		2.39	1341.7
11_3_2013 siloxanes sludge 01ngmL 1	Analyte		2.39	1806.0
11_3_2013 siloxanes sludge 05ngmL 1	Analyte		2.39	2067.4
11_3_2013 siloxanes sludge 1 ngmL 1	Analyte		2.39	2935.6
11_3_2013 siloxanes sludge 5 ngmL 1	Analyte		2.39	9014.2
11_3_2013 siloxanes sludge 10 ngmL 1	Analyte		2.39	16739.1
11_3_2013 siloxanes sludge 50 ngmL 1	Analyte		2.39	109751.0
11_3_2013 siloxanes sludge blank 2	Blank		2.39	1520.2
11_3_2013 siloxanes sludge 01ngmL 2	Analyte		2.39	1805.6
11_3_2013 siloxanes sludge 05ngmL 2	Analyte		2.39	2567.9
11_3_2013 siloxanes sludge 1 ngmL 2	Analyte		2.39	2880.1
11_3_2013 siloxanes sludge 5 ngmL 2	Analyte		2.39	9802.0
11_3_2013 siloxanes sludge 10 ngmL 2	Analyte		2.39	14716.8
11_3_2013 siloxanes sludge 50 ngmL 2	Analyte		2.39	116916.0
11_3_2013 siloxanes sludge blank 3	Blank		2.38	1702.1
11_3_2013 siloxanes sludge 01ngmL 3	Analyte		2.38	2036.9
11_3_2013 siloxanes sludge 05ngmL 3	Analyte		2.38	2368.3
11_3_2013 siloxanes sludge 1 ngmL 3	Analyte		2.38	3046.0
11_3_2013 siloxanes sludge 5 ngmL 3	Analyte		2.38	9017.0
11_3_2013 siloxanes sludge 10 ngmL 3	Analyte		2.38	16719.0
11_3_2013 siloxanes sludge 50 ngmL 3	Analyte		2.38	114432.3

Table A-23: Data of D4 QAQC analysis with split ratio 1:10 on 11/4/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
11_4_2013 siloxanes water blank1	Blank		2.38	72.7
11_4_2013 siloxanes water 01ngmL 1	Standard	0.1	2.38	1347.2
11_4_2013 siloxanes water 05ngmL 1	Standard	0.5	2.38	2878.4
11_4_2013 siloxanes water 1 ngmL 1	Standard	1	2.38	4269.0
11_4_2013 siloxanes water 5 ngmL 1	Standard	5	2.38	28550.9

11_4_2013 siloxanes water 10 ngmL 1	Standard	10	2.38	44408.1
11_4_2013 siloxanes water 50 ngmL 1	Standard	50	2.38	276218.8
11_4_2013 siloxanes water blank 2	Blank		2.38	232.5
11_4_2013 siloxanes water 01ngmL 2	Standard	0.1	2.38	1124.5
11_4_2013 siloxanes water 05ngmL 2	Standard	0.5	2.38	2407.5
11_4_2013 siloxanes water 1 ngmL 2	Standard	1	2.38	3684.7
11_4_2013 siloxanes water 5 ngmL 2	Standard	5	2.38	20914.3
11_4_2013 siloxanes water 10 ngmL 2	Standard	10	2.38	40475.9
11_4_2013 siloxanes water 50 ngmL 2	Standard	50	2.38	257189.1
11_4_2013 siloxanes water blank 3	Blank		2.38	190.0
11_4_2013 siloxanes water 01ngmL 3	Standard	0.1	2.38	1072.2
11_4_2013 siloxanes water 05ngmL 3	Standard	0.5	2.38	2196.3
11_4_2013 siloxanes water 1 ngmL 3	Standard	1	2.38	3601.8
11_4_2013 siloxanes water 5 ngmL 3	Standard	5	2.38	21598.0
11_4_2013 siloxanes water 10 ngmL 3	Standard	10	2.38	36445.7
11_4_2013 siloxanes water 50 ngmL 3	Standard	50	2.38	237337.1
11_4_2013 siloxanes sludge blank1	Blank		2.38	1147.8
11_4_2013 siloxanes sludge 01ngmL 1	Analyte		2.38	1324.7
11_4_2013 siloxanes sludge 05ngmL 1	Analyte		2.38	1719.2
11_4_2013 siloxanes sludge 1 ngmL 1	Analyte		2.38	2412.5
11_4_2013 siloxanes sludge 5 ngmL 1	Analyte		2.38	8439.9
11_4_2013 siloxanes sludge 10 ngmL 1	Analyte		2.38	13842.9
11_4_2013 siloxanes sludge 50 ngmL 1	Analyte		2.38	66127.9
11_4_2013 siloxanes sludge blank 2	Blank		2.38	1194.8
11_4_2013 siloxanes sludge 01ngmL 2	Analyte		2.38	1614.2
11_4_2013 siloxanes sludge 05ngmL 2	Analyte		2.38	1844.6
11_4_2013 siloxanes sludge 1 ngmL 2	Analyte		2.38	2568.9
11_4_2013 siloxanes sludge 5 ngmL 2	Analyte		2.38	7202.0
11_4_2013 siloxanes sludge 10 ngmL 2	Analyte		2.38	12787.0
11_4_2013 siloxanes sludge 50 ngmL 2	Analyte		2.38	63593.7
11_4_2013 siloxanes sludge blank 3	Blank		2.38	1286.5
11_4_2013 siloxanes sludge 01ngmL 3	Analyte		2.38	1836.9
11_4_2013 siloxanes sludge 05ngmL 3	Analyte		2.38	2020.6
11_4_2013 siloxanes sludge 1 ngmL 3	Analyte		2.38	2813.8
11_4_2013 siloxanes sludge 5 ngmL 3	Analyte		2.38	7058.0
11_4_2013 siloxanes sludge 10 ngmL 3	Analyte		2.38	13436.6
11_4_2013 siloxanes sludge 50 ngmL 3	Analyte		2.38	87196.7

Table A-24: Data of D4 Fort Collins and Loveland analysis with split ratio 1:10 on 11/16/13

Name	Type	Std. Conc	RT	Area
2013_11_16 Siloxanes water Blank 1	Water Blank	0	2.38	322.2
2013_11_16 Siloxanes water 0_1ngmL	Water Standard	0.5	2.38	1816.9
2013_11_16 Siloxanes water 5 ngmL	Water Standard	5	2.38	33400.8
2013_11_16 Siloxanes water 50 ngmL	Water Standard	50	2.38	387701.3
2013_11_16 Siloxanes sludge Blank 2	Sludge Blank	0	2.38	1064.5
2013_11_16 Siloxanes sludge 0_1 ngmL	Sludge Standard	0.1	2.39	1239.9
2013_11_16 Siloxanes sludge 0_5 ngmL	Sludge Standard	0.5	2.38	2560.6
2013_11_16 Siloxanes sludge 1 ngmL	Sludge Standard	1	2.38	3044.5
2013_11_16 Siloxanes sludge 5 ngmL	Sludge Standard	5	2.39	13660.6
2013_11_16 Siloxanes sludge 10 ngmL	Sludge Standard	10	2.38	34777.7
2013_11_16 Siloxanes sludge 50 ngmL	Sludge Standard	50	2.38	169758.7
2013_11_16 Siloxanes water Blank 3	Blank		2.38	150.1
2013_11_16 Siloxanes FC 11 Effluent	Water Analyte		2.38	179.8
2013_11_16 Siloxanes FC 21 Influent	Water Analyte		2.38	469.2
2013_11_16 Siloxanes FC 31 1sludge	Sludge Analyte		2.38	4072.6
2013_11_16 Siloxanes FC 41 WAS	Sludge Analyte		2.39	1645.1
2013_11_16 Siloxanes FC 51 Digested sludge	Sludge Analyte		2.38	1099.2
2013_11_16 Siloxanes water Blank 4	Blank		2.39	98.1
2013_11_16 Siloxanes FC 12 Effluent	Water Analyte		2.39	302.9
2013_11_16 Siloxanes FC 22 Influent	Water Analyte		2.38	650.4
2013_11_16 Siloxanes FC 32 1sludge	Sludge Analyte		2.38	4240.3
2013_11_16 Siloxanes FC 42 WAS	Sludge Analyte		2.38	1641.3
2013_11_16 Siloxanes FC 52 Digested sludge	Sludge Analyte		2.38	1203.6
2013_11_16 Siloxanes water Blank 5	Blank		2.38	113.2
2013_11_16 Siloxanes LL 11 Effluent	Water Analyte		2.38	225.8
2013_11_16 Siloxanes LL 21 PEF	Water Analyte		2.38	437.5
2013_11_16 Siloxanes LL 31 Primary Effluent	Water Analyte		2.38	433.8
2013_11_16 Siloxanes LL 41 Influent	Water Analyte		2.38	452.3
2013_11_16 Siloxanes LL 51 2 digester	Sludge Analyte		2.38	777.5
2013_11_16 Siloxanes LL 61 1 digester	Sludge Analyte		2.38	1413.0
2013_11_16 Siloxanes LL 71 Primary sludge	Sludge Analyte		2.38	2656.7
2013_11_16 Siloxanes LL 81 WAS	Sludge Analyte		2.38	349.4
2013_11_16 Siloxanes water Blank 6	Blank		2.38	93.0
2013_11_16 Siloxanes LL 12 Effluent	Water Analyte		2.38	254.6

2013_11_16 Siloxanes LL 22 PEF	Water Analyte		2.38	499.5
2013_11_16 Siloxanes LL 32 Primary Effluent	Water Analyte		2.38	473.4
2013_11_16 Siloxanes LL 42 Influent	Water Analyte		2.38	506.3
2013_11_16 Siloxanes LL 52 2 digester	Sludge Analyte		2.38	640.5
2013_11_16 Siloxanes LL 62 1 digester	Sludge Analyte		2.38	1551.7
2013_11_16 Siloxanes LL 72 Primary sludge	Sludge Analyte		2.38	3695.7
2013_11_16 Siloxanes LL 82 WAS	Sludge Analyte		2.38	341.0

Table A-25: Data of D4 Boulder analysis with split ratio 1:10 on 11/22/13

Name	Type	Std. Conc	RT	Area
2013_11_22 Siloxanes water Blank 1	Blank			
2013_11_22 Siloxanes water 0_1ngmL	Standard	0.5	2.34	1521.2
2013_11_22 Siloxanes water 5 ngmL	Standard	5	2.34	31312.7
2013_11_22 Siloxanes water 50 ngmL	Standard	50	2.34	270118.1
2013_11_22 Siloxanes sludge Blank 2	Blank		2.35	2491.9
2013_11_22 Siloxanes sludge 0_1 ngmL	Standard	0.1	2.35	2705.9
2013_11_22Siloxanes sludge 0_5 ngmL	Standard	0.5	2.34	3851.3
2013_11_22 Siloxanes sludge 1 ngmL	Standard	1	2.35	4439.6
2013_11_22 Siloxanes sludge 5 ngmL	Standard	5	2.35	11054.1
2013_11_22 Siloxanes sludge 10 ngmL	Standard	10	2.35	23236.8
2013_11_22 Siloxanes sludge 50 ngmL	Standard	50	2.35	104041.3
2013_11_22 Siloxanes water Blank 3	Blank		2.34	133.1
2013_11_22 Siloxanes water Blank 4	Blank		2.35	139.3
2013_11_22 Siloxanes water Blank 5	Blank		2.35	124.1
2013_11_22 Siloxanes Rec 0_25ngmL1	Analyte		2.35	2633.1
2013_11_22 Siloxanes Rec 0_25ngmL2	Analyte		2.35	2388.7
2013_11_22 Siloxanes Rec 0_25ngmL3	Analyte		2.35	2636.6
2013_11_22 Siloxanes Rec 7_5 ngmL1	Analyte		2.35	16892.8
2013_11_22 Siloxanes Rec 7_5 ngmL2	Analyte		2.35	13051.2
2013_11_22 Siloxanes Rec 7_5 ngmL3	Analyte		2.35	14819.1
2013_11_22 Siloxanes Rec 25 ngmL 1	Analyte		2.35	44614.1
2013_11_22 Siloxanes Rec 25 ngmL 2	Analyte		2.35	50442.0
2013_11_22 Siloxanes Rec 25 ngmL 3	Analyte		2.35	50411.5
2013_11_22 Siloxanes BD Effluent 1	Analyte		2.34	148.1
2013_11_22 Siloxanes BD Effluent 2	Analyte		2.35	126.4
2013_11_22 Siloxanes BD Centrifuge 1	Analyte		2.34	150.1
2013_11_22 Siloxanes BD Centrifuge 2	Analyte		2.34	157.7
2013_11_22 Siloxanes BD Influent 1	Analyte		2.34	284.4

2013_11_22 Siloxanes BD Influent 2	Analyte		2.34	231.6
2013_11_22 Siloxanes BD Digester 1	Analyte		2.34	511.9
2013_11_22 Siloxanes BD Digester 2	Analyte		2.35	530.8
2013_11_22 Siloxanes BD WAS 1	Analyte		2.35	3688.8
2013_11_22 Siloxanes BD WAS 2	Analyte		2.35	3208.8
2013_11_22 Siloxanes Primary sludge 1	Analyte		2.35	2488.8
2013_11_22 Siloxanes Primary sludge 2	Analyte		2.35	2129.0

Table A-26: Data of D4 Fort Collins and Loveland analysis with split ratio 1:10 on 1/10/2014

Name	Type	Std. Conc	RT	Area
2014_1_10 siloxane blank1 water	Blank			
2014_1_10 siloxane 05D405D5water	Standard	0.5	2.36	1823.7
2014_1_10 siloxane 1D45D5 water	Standard	1	2.37	2748.8
2014_1_10 siloxane 5D450D5water	Standard	5	2.37	9802.6
2014_1_10 siloxane 15D4150D5 water	Standard	15	2.36	35586.9
2014_1_10 siloxane blank2 sludge	Blank		2.37	10784.7
2014_1_10 siloxane 05D405D5sludge	Standard	0.5	2.37	11740.7
2014_1_10 siloxane 1D45D5 sludge	Standard	1	2.37	8574.3
2014_1_10 siloxane 5D450D5sludge	Standard	5	2.37	14249.5
2014_1_10 siloxane 15D4150D5 sludge	Standard	15	2.37	22313.8
2014_1_10 siloxane blank3 water	Blank		2.37	385.5
2014_1_10 siloxane blank4 water	Blank		2.37	290.2
2014_1_10 siloxane blank5 water	Blank		2.37	476.4
2014_1_10 siloxane blank6 water	Blank		2.37	442.3
2014_1_10 siloxane FC effluent1	Analyte		2.37	554.0
2014_1_10 siloxane FC effluent2	Analyte		2.37	376.1
2014_1_10 siloxane FCinfluent1	Analyte		2.37	1079.2
2014_1_10 siloxane FCinfluent2	Analyte		2.37	1124.2
2014_1_10 siloxane FCcentrate1	Analyte		2.37	561.7
2014_1_10 siloxane FCcentrate2	Analyte		2.37	555.3
2014_1_10 siloxane FC primary sludge1	Analyte		2.37	17551.9
2014_1_10 siloxane FC primary sludge2	Analyte		2.37	11606.5
2014_1_10 siloxane FC WAS1	Analyte		2.37	30366.8
2014_1_10 siloxane FC WAS2	Analyte		2.37	25228.6
2014_1_10 siloxane FC RAS 1	Analyte		2.37	5347.4
2014_1_10 siloxane FC RAS 2	Analyte		2.37	4847.1
2014_1_10 siloxane FC anaerobic sludge1	Analyte		2.37	4211.6
2014_1_10 siloxane FC anaerobic sludge2	Analyte		2.37	5287.1
2014_1_10 siloxane LL effluent 1	Analyte		2.37	653.9

2014_1_10 siloxane LL effluent 2	Analyte		2.37	947.1
2014_1_10 siloxane LL primary effluent 1	Analyte		2.37	944.4
2014_1_10 siloxane LL primary effluent 2	Analyte		2.37	1045.6
2014_1_10 siloxane LL influent 1	Analyte		2.37	969.1
2014_1_10 siloxane LL influent 2	Analyte		2.37	942.4
2014_1_10 siloxane LL Centrate 1	Analyte		2.37	1078.0
2014_1_10 siloxane LL Centrate 2	Analyte		2.36	1087.8
2014_1_10 siloxane LL primary digester 1	Analyte		2.37	3680.6
2014_1_10 siloxane LL primary digester 2	Analyte		2.37	3934.3
2014_1_10 siloxane LL RAS 1	Analyte		2.37	1241.0
2014_1_10 siloxane LL RAS 2	Analyte		2.37	1630.3
2014_1_10 siloxane LL Primary sludge 1	Analyte		2.37	5727.3
2014_1_10 siloxane LL Primary sludge 2	Analyte		2.37	5016.5

Table A-27: Data of D4 Fort Collins and Loveland analysis with split ratio 1:10 on 2/5/2014

Name	Type	Std. Conc	RT	Area
2014_2_5 siloxane blank1 water	Blank		2.35	1348.1
2014_2_5 siloxane 05D405D5water	Standard	0.5	2.35	2485.0
2014_2_5 siloxane 1D45D5 water	Standard	1	2.35	3498.4
2014_2_5 siloxane 5D450D5water	Standard	5	2.35	10154.3
2014_2_5 siloxane 15D4150D5 water	Standard	15	2.35	29932.8
2014_2_5 siloxane blank2 sludge	Blank		2.35	7452.9
2014_2_5 siloxane 05D405D5sludge	Standard	0.5	2.35	7904.2
2014_2_5 siloxane 1D45D5 sludge	Standard	1	2.35	8481.1
2014_2_5 siloxane 5D450D5sludge	Standard	5	2.35	13068.3
2014_2_5 siloxane 15D4150D5 sludge	Standard	15	2.35	24733.5
2014_2_5 siloxane blank3 water	Blank		2.35	870.6
2014_2_5 siloxane blank4 water	Blank		2.35	856.6
2014_2_6 siloxanes Blank water 5	Blank		2.36	1971.0
2014_2_5 siloxane Blank sludge	Blank		2.35	3245.6
2014_2_5 siloxane LL effluent 1	Analyte		2.35	945.1
2014_2_5 siloxane LL effluent 2	Analyte		2.35	982.5
2014_2_5 siloxane LL primary effluent 1	Analyte		2.35	1387.6
2014_2_5 siloxane LL primary effluent 2	Analyte		2.35	1449.9
2014_2_5 siloxane LL influent 1	Analyte		2.35	1202.6
2014_2_5 siloxane LL influent 2	Analyte		2.35	1293.4
2014_2_5 siloxane LL Centrate 1	Analyte		2.35	2201.1
2014_2_5 siloxane LL Centrate 2	Analyte		2.35	2441.1
2014_2_5 siloxane LL primary digester 1	Analyte		2.35	2132.5

2014_2_5 siloxane LL primary digester 2	Analyte		2.35	2256.1
2014_2_5 siloxane LL primary sludge 1	Analyte		2.36	8837.7
2014_2_5 siloxane LL Primary sludge 2	Analyte		2.35	8930.9
2014_2_5 siloxane LL secondary sludge 1	Analyte		2.35	1616.0
2014_2_5 siloxane LL secondary sludge2	Analyte		2.35	1546.8
2014_2_5 siloxane LL Primary clarifier 1	Analyte		2.35	1360.9
2014_2_5 siloxane LL Primary clarifier 2	Analyte		2.35	1456.0
2014_2_6 siloxanes FC eff 1	Analyte		2.36	714.6
2014_2_6 siloxanes FC eff 2	Analyte		2.36	701.6
2014_2_6 siloxanes FC inf 1	Analyte		2.36	1317.2
2014_2_6 siloxanes FC inf 2	Analyte		2.36	1238.8
2014_2_6 siloxanes FC Cen 1	Analyte		2.36	620.3
2014_2_6 siloxanes FC Cen 2	Analyte		2.36	633.1
2014_2_6 siloxanes FC Pri sludge 1	Analyte		2.36	6149.5
2014_2_6 siloxanes FC Pri sludge 2	Analyte		2.36	6104.6
2014_2_6 siloxanes FC TWAS 1	Analyte		2.36	6409.2
2014_2_6 siloxanes FC TWAS 2	Analyte		2.36	6897.3
2014_2_6 siloxanes FC RAS 1	Analyte		2.36	3476.6
2014_2_6 siloxanes FC RAS 2	Analyte		2.36	3821.2
2014_2_6 siloxanes FC Anaerobic Sludge 1	Analyte		2.36	1674.8
2014_2_6 siloxanes FC Anaerobic Sludge 2	Analyte		2.37	1915.0

Table A-28: Data of D4 Fort Collins and Loveland analysis with split ratio 1:10 on 3/5/2014

Name	Type	Std. Conc	RT	Area
2014_3_5 siloxanes Blank 1	Blank	0	2.35	813.6
2014_3_5 siloxanes water 05D405D5	Standard	0.5	2.35	1230.2
2014_3_5 siloxanes water 1D45D5	Standard	1	2.35	1658.2
2014_3_5 siloxanes water 5D450D5	Standard	5	2.35	20797.5
2014_3_5 siloxanes water15D4150D5	Standard	15	2.35	69290.1
2014_3_5 siloxanes sludge Blank 2	Blank	0	2.35	4964.8
2014_3_5 siloxanes sludge 05D405D5	Standard	0.5	2.35	5060.6
2014_3_5 siloxanes sludge 1D45D5	Standard	1	2.35	5206.3
2014_3_5 siloxanes sludge 5D450D5	Standard	5	2.35	5931.2
2014_3_5 siloxanes sludge15D4150D5	Standard	15	2.35	8291.5
2014_3_5 siloxanes water Blank 3	Blank	0	2.35	257.6
2014_3_5 siloxanes water Blank 4	Blank	0	2.35	174.4
2014_3_6 siloxanes FC water blank1	Blank		2.36	120.8
2014_3_6 siloxanes FC water blank 2	Blank		2.36	188.9
2014_3_5 siloxanes LL effluent1	Analyte		2.35	255.7

2014_3_5 siloxanes LL effluent 2	Analyte		2.35	338.9
2014_3_5 siloxanes LL PEF 1	Analyte		2.35	598.7
2014_3_5 siloxanes LL PEF 2	Analyte		2.35	536.8
2014_3_5 siloxanes LL Influent 1	Analyte		2.35	546.7
2014_3_5 siloxanes LL Influent 2	Analyte		2.35	490.4
2014_3_5 siloxanes LL RAS 1	Analyte		2.35	594.5
2014_3_5 siloxanes LL RAS 2	Analyte		2.35	633.1
2014_3_5 siloxanes LL Primary Digester 1	Analyte		2.35	1257.1
2014_3_5 siloxanes LL Primary Digester 2	Analyte		2.35	1192.1
2014_3_5 siloxanes LL Primary sludge 1	Analyte		2.36	3896.2
2014_3_5 siloxanes LL Primary sludge 2	Analyte		2.35	3139.6
2014_3_6 siloxanes FC effluent 1	Analyte		2.36	177.5
2014_3_6 siloxanes FC effluent 2	Analyte		2.36	160.5
2014_3_6 siloxanes FC influent 1	Analyte		2.36	394.6
2014_3_6 siloxanes FC influent 2	Analyte		2.36	448.0
2014_3_6 siloxanes FC centrate 1	Analyte			
2014_3_6 siloxanes FC centrate 2	Analyte		2.36	238.4
2014_3_6 siloxanes FC Primary sludge 1	Analyte		2.36	3442.5
2014_3_6 siloxanes FC Primary sludge 2	Analyte		2.36	3484.1
2014_3_6 siloxanes FC TWAS 1	Analyte		2.36	2959.7
2014_3_6 siloxanes FC TWAS 2	Analyte		2.36	2630.8
2014_3_6 siloxanes FC RAS 1	Analyte		2.36	1616.8
2014_3_6 siloxanes FC RAS 2	Analyte		2.36	1186.3
2014_3_6 siloxanes FC Anaerobic sludge 1	Analyte		2.36	889.2
2014_3_6 siloxanes FC Anaerobic sludge 2	Analyte		2.36	882.6

Table A-29: Data of D4 Fort Collins and Loveland analysis with split ratio 1:10 on 4/16/2014

Name	Type	Std. Conc	RT	Area
2014_4_16 siloxanes water 05D405D5	Standard	0.5	2.35	691.146
2014_4_16 siloxanes water 1D45D5	Standard	1	2.34	826.321
2014_4_16 siloxanes water 5D450D5	Standard	5	2.34	1590.977
2014_4_16 siloxanes water 15D4150D5	Standard	15	2.34	4457.479
2014_4_16 siloxanes sludge 05D405D5	Standard	0.5	2.35	1182.401
2014_4_16 siloxanes sludge 1D45D5	Standard	1	2.34	1261.944
2014_4_16 siloxanes sludge 5D450D5	Standard	5	2.34	1877.327
2014_4_16 siloxanes sludge 15D4150D5	Standard	15	2.34	3714.612

2014_4_16 siloxanes blank water 1	Blank		2.34	557.962
2014_4_16 silxoanes blank sludge 2	Blank		2.35	1071.547
2014_4_16 siloxanes Water blank 3	Blank		2.34	246.272
2014_4_16 siloxanes Water blank 4	Blank		2.34	124.864
2014_4_16 siloxanes water blank 5	Blank		2.34	167.278
2014_4_16 siloxanes water blank 6	Blank		2.34	101.525
2014_4_16 siloxanes FC effluent 1	Analyte		2.34	427.71
2014_4_16 siloxanes FC effluent 2	Analyte		2.34	274.461
2014_4_16 siloxanes FC Influent 1	Analyte		2.34	564.531
2014_4_16 siloxanes FC Influent 2	Analyte		2.34	448.519
2014_4_16 siloxanes FC Centrate 1	Analyte		2.34	309.954
2014_4_16 siloxanes FC Centrate 2	Analyte		2.34	347.59
2014_4_16 siloxanes FC Primary sludge 1	Analyte		2.34	1947.507
2014_4_16 siloxanes FC Primary sludge 2	Analyte		2.34	2319.757
2014_4_16 siloxanes FC TWAS 1	Analyte		2.34	2378.492
2014_4_16 siloxanes FC TWAS 2	Analyte		2.34	1883.147
2014_4_16 siloxanes FC RAS 1	Analyte		2.34	1172.204
2014_4_16 siloxanes FC RAS 2	Analyte		2.34	1257.231
2014_4_16 siloxanes FC Anaerobic sludge 1	Analyte		2.34	721.405
2014_4_16 siloxanes FC Anaerobic sludge 2	Analyte		2.34	640.58
2014_4_16 siloxanes LL effluent 1	Analyte		2.33	325.696
2014_4_16 siloxanes LL effluent 2	Analyte		2.34	348.583
2014_4_16 siloxanes LL primary effluent 1	Analyte		2.34	400.849
2014_4_16 siloxanes LL primary effluent 2	Analyte		2.34	428.314
2014_4_16 siloxanes LL influent 1	Analyte		2.34	323.755
2014_4_16 siloxanes LL influent 2	Analyte		2.34	315.311
2014_4_16 siloxanes LL Centrate 1	Analyte		2.34	163.076
2014_4_16 siloxanes LL Centrate 2	Analyte		2.34	156.865
2014_4_16 siloxanes LL RAS1	Analyte		2.34	524.887
2014_4_16 siloxanes LL RAS 2	Analyte		2.34	423.629
2014_4_16 siloxanes LL Primary digestion 1	Analyte		2.34	513.582
2014_4_16 siloxanes LL Primary digestion 2	Analyte		2.34	539.185
2014_4_16 siloxanes LL primary sludge 1	Analyte		2.34	1511.772
2014_4_16 siloxanes LL primary sludge 2	Analyte		2.34	1947.245

Table A-30: Data of D4 Fort Collins and Loveland analysis with split ratio 1:10 on 5/20/2014

Name	Type	Std. Conc	RT	Area
2014_5_20 siloxane blank water 1	Blank		2.33	994.527
2014_5_20 siloxane water 0505ngmL	Standard	0.5	2.33	1730.189
2014_5_20 siloxane water D41D55ngmL	Standard	1	2.33	4164.804
2014_5_20 siloxane water D45D550ngmL	Standard	5	2.33	14711.1

2014_5_20 siloxane water D415D5150ngmL	Standard	15	2.33	50817.95
2014_5_21 siloxanes sludge blank	Blank		2.32	4072.808
2014_5_20 siloxane sludge D405D505ngmL	Standard	0.5	2.33	5349.353
2014_5_20 siloxane sludge D41D55ngmL	Standard	1	2.33	6086.096
2014_5_20 siloxane sludge D45D550ngmL	Standard	5	2.33	13244.86
2014_5_20 siloxane sludge D415D5150ngmL	Standard	15	2.33	30277.44
2014_5_20 siloxane blank water 00	Blank		2.33	458.269
2014_5_20 siloxane blank water 2	Blank		2.33	766.533
2014_5_20 siloxane blank water 3	Blank		2.33	669.557
2014_5_20 siloxane blank water 4	Blank		2.33	420.92
2014_5_20 siloxane blank water 5	Blank		2.33	753.852
2014_5_20 siloxane FC Effluent 1	Analyte		2.33	821.769
2014_5_20 siloxane FC Effluent 2	Analyte		2.33	1101.293
2014_5_20 siloxane FC influent 1	Analyte		2.33	1648.86
2014_5_20 siloxane FC influent 2	Analyte		2.33	1090.595
2014_5_20 siloxane FC centrate 1	Analyte		2.33	634.819
2014_5_20 siloxane FC centrate 2	Analyte		2.33	916.731
2014_5_20 siloxane FC primary sludge 1	Analyte		2.33	10819.31
2014_5_20 siloxane FC primary sludge 2	Analyte		2.33	11145.2
2014_5_20 siloxane FC TWAS 1	Analyte		2.33	12731.02
2014_5_20 siloxane FC TWAS 2	Analyte		2.33	15121.65
2014_5_20 siloxane FC RAS 1	Analyte		2.33	4682.845
2014_5_20 siloxane FC RAS 2	Analyte		2.33	4775.412
2014_5_20 siloxane FC Digester 1	Analyte		2.33	2968.536
2014_5_20 siloxane FC Digester 2	Analyte		2.33	3644.502
2014_5_20 siloxane LL Effluent 1	Analyte		2.33	920.152
2014_5_20 siloxane LL Effluent 2	Analyte		2.32	1005.719
2014_5_20 siloxane LL Primary effluent 1	Analyte		2.33	1614.237
2014_5_20 siloxane LL Primary effluent 2	Analyte		2.32	1542.313
2014_5_20 siloxane LL influent 1	Analyte		2.33	1373.952
2014_5_20 siloxane LL influent 2	Analyte		2.33	1241.041
2014_5_20 siloxane LL Centrate 1	Analyte		2.33	1378.201
2014_5_20 siloxane LL Centrate 2	Analyte		2.32	1541.489
2014_5_20 siloxane LL RAS 1	Analyte		2.33	8582.556
2014_5_20 siloxane LL RAS 2	Analyte		2.32	8164.465
2014_5_20 siloxane LL Digester 1	Analyte		2.33	2972.656
2014_5_20 siloxane LL Digester 2	Analyte		2.32	3403.399
2014_5_20 siloxane LL Primary sludge 1	Analyte		2.33	10172.01
2014_5_20 siloxane LL Primary sludge 2	Analyte		2.32	10250.8

Table A-31: Data of D4 Volume of Acetone Impact on 5/20/2014

Name	Type	Std.	RT	Area

		Conc		
2014_5_20 siloxane acetone 50uL water	Analyte		2.33	3013.688
2014_5_20 siloxane acetone 100uL water	Analyte		2.32	5050.129
2014_5_20 siloxane acetone 500uL water	Analyte		2.28	9431.552
2014_5_20 siloxane acetone 1000uL water	Analyte		2.31	14351.71
2014_5_20 siloxane acetone 1500uL water	Analyte		2.27	2982.712
2014_5_20 siloxane acetone 1500uL water2	Analyte		2.27	5724.343
2014_5_20 siloxane acetone 50uL sludge	Analyte		2.33	8108.393
2014_5_20 siloxane acetone 100uL sludge	Analyte		2.33	6628.388
2014_5_20 siloxane acetone 500uL sludge	Analyte		2.31	15934.18
2014_5_20 siloxane acetone 1000uL sludge	Analyte		2.29	23701.99
2014_5_20 siloxane acetone 1500uL sludge	Analyte		2.3	33790.42
2014_5_20 siloxane acetone 1500uL sludge2	Analyte		2.3	35033.05

B. Raw Data – D5

Table B-1: Data of D5 during feasibility test on 4/4/13

Name	Type	Std. Conc (ng·mL^{-1})	RT	Area
4_4_13 D4 D5 Standard	Analyte	50	6.84	567509.1
4_4_13 Sludge	Analyte		6.84	47845.0
4_4_13 Sluge + 3ug	Analyte		6.84	51163.5
4_4_13 Sludge + 10ug	Analyte		6.84	249510.5
4_4_13 3ug std	Standard	150	6.85	1781782.9
4_4_13 10ug std	Standard	500	6.84	5104309.0

Table B-2: Data of D5 analysis on 5/7/13

Name	Type	Std. Conc (ng·mL^{-1})	RT	Area
05-07-13_siloxane_0	Standard	0	6.86	1500.1
05-07-13_siloxane_25ngml	Standard	25	6.86	2596.1
05-07-13_siloxane_100ngml	Standard	100	6.86	994.6
05-07-13_siloxane_250ngml	Standard	250	6.86	34179.8
05-07-13_siloxane_0_sludge1	Analyte		6.86	3019.4
05-07-13_siloxane_0_sludge2	Analyte		6.86	6061.6
05-07-13siloxane_25_sludge1	Analyte	25	6.86	2414.9
05-07-13siloxane_25_sludge2	Analyte	25	6.86	6450.5
05-07-13siloxane_50_sludge1	Analyte	50	6.86	36827.7
05-07-13siloxane_50_sludge2	Analyte	50	6.86	27193.1
05-07-13siloxane_75_sludge1	Analyte	75	6.86	23805.0
05-07-13siloxane_75_sludge2	Analyte	75	6.86	60330.5
05-07-13siloxane_100_sludge1	Analyte	100	6.86	1642.1
05-07-13siloxane_100_sludge2	Analyte	100	6.86	1820.2
05-07-13siloxane_250_sludge1	Analyte	250	6.86	2295.2
05-07-13siloxane_250_sludge2	Analyte	250	6.86	2559.3

Table B-3: Data of D5 analysis on 7/12/13

Name	Type	Std. Conc (ng·mL^{-1})	RT	Area
7_12_13_Siloxane_Sample 1	Analyte		6.92	67.4
7_12_13_Siloxane_Sample 2	Analyte		6.91	149.5
7_12_13_Siloxane_0	Blank		6.51	38.3

7_12_13_Siloxane_50ngmL	Standard	50	6.92	191.6
7_12_13_Siloxane_100ngmL	Standard	100		
7_12_13_Siloxane_500ngmL	Standard	500	6.93	84.5

Table B-4: Data of D5 analysis for Fort Collins and Loveland on 7/18/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
7_18_13_Siloxane_Blank1	Blank		2.32	515.3
7_18_13_Siloxane_Post1	Analyte		2.33	10045.7
7_18_13_Siloxane_Post2	Analyte		2.33	11147.2
7_18_13_Siloxane_Pre1	Analyte		2.33	11567.3
7_18_13_Siloxane_Pre2	Analyte		2.33	13702.7
7_18_13_Siloxane_LL1	Analyte		2.33	9517.9
7_18_13_Siloxane_LL2	Analyte		2.32	10250.3
7_18_13_Siloxane_Blank2	Blank	0	2.32	55.5
7_18_13_Siloxane_5ngmL	Standard	5	2.32	1381.5
7_18_13_Siloxane_10ngmL	Standard	10	2.33	2879.6
7_18_13_Siloxane_25ngml	Standard	25	2.33	6944.5
7_18_13_Siloxane_Blank3	Blank		2.33	159.6

Table B-5: Data of D5 analysis on 9/4/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_4_13 Siloxane_Blank1	Blank		5.64	409.6
9_4_13 Siloxane_1ngml	Standard	10	5.64	273.4
9_4_13 Siloxane_2_5ngml	Standard	25	5.64	245.8
9_4_13 Siloxane_7_5ngml	Standard	75	5.64	207.9
9_4_13 Siloxane_Blank2	Blank		5.64	268.1
9_4_13 Siloxane_sludge1	Analyte		5.64	104.1
9_4_13 Siloxane_sludge2	Analyte		5.64	106.8
9_4_13 Siloxane_Blank3	Blank		5.64	126.4

Table B-6: Data of D5 analysis on 9/10/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_10_13 Siloxane	Blank		5.75	82.9
9_10_13 Siloxane_blank1	Blank		5.63	160.6
9_10_13 Siloxane1ngml	Standard	10	5.63	24.6
9_10_13 Siloxane2_5ngml	Standard	25	5.63	80.4

Table B-7: Data of D5 during blank test on 9/11/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_11_2013 Acetone	Blank		5.76	21.7
9_11_2013 Water	Blank		5.76	137.1
9_11_2013 IS	Analyte		5.63	19.7

Table B-8: Data of D5 analysis repeated on 9/11/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_11_2013 Siloxaneblank	Blank	0		
9_11_2013 Siloxane1ngml	Standard	10	5.63	47.1
9_11_2013 Siloxane25ngml	Standard	25	5.63	18.8
9_11_2013 Siloxane75ngml	Standard	75	5.63	32.1

Table B-9: Data of D5 analysis on 9/12/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_12_2013 Siloxaneblank	Blank	0	5.76	28.4
9_12_2013 Siloxane1ngml	Standard	10	5.76	46.4
9_12_2013 Siloxane2_5ngml	Standard	25	5.76	36.9
9_12_2013 Siloxane7_5ngml	Standard	75	5.76	52.2

Table B-10: Data of D5 in water analysis on 9/30/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
2013_9_30Siloxans_water 1	Standard	10	2.41	240070.6
2013_9_30Siloxans_water 2	Standard	50	2.41	913027.2
2013_9_30Siloxans_water 3	Standard	100	2.41	2261197.0

Table B-11: Data of D5 in sludge analysis on 9/30/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
2013_9_30Siloxans_sludge 1	Blank	0	2.41	65198.2
2013_9_30Siloxans_sludge 2	Standard	10	2.41	115404.0
2013_9_30Siloxans_sludge 3	Standard	50	2.42	303220.2
2013_9_30Siloxans_sludge 4	Standard	100	2.42	631180.2

Table B-12: Data of D5 analysis on 10/1/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
2013_10_1 Siloxane 0ngmL	Blank	0	2.41	60735.3
2013_10_1 Siloxane 10ngmL	Analyte	10	2.41	124413.7

Table B-13: Data of D5 analysis with initial temperature of 60 ℃ on 10/4/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_4_2013 Siloxanes IS 60 C	Blank		3.55	107470.3

Table B-14: Data of D5 analysis with initial temperature of 70 ℃ on 10/4/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_4_2013 Siloxane full scan	Analyte	25		
10_4_2013 Siloxane IS with 70C	Analyte	50	3.17	408466.3

Table B-15: Data of D5 analysis with initial temperature of 80 ℃ on 10/7/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_7_13 Siloxane at 80C		50	2.9	115570.1

Table B-16: Data of D5 analysis with initial temperature of 75 ℃ on 10/7/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_7_13 Siloxane at 75C			3.18	143229.9

Table B-17: Data of D5 QAQC analysis with split ratio of 1: 20 on 10/8/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_8_13 slixane QAQC Blank1	Blank	0	3.19	869.7
10_8_13 slixane QAQC 10ngmLwater1	Standard	10	3.2	88654.8
10_8_13 slixane QAQC 100ngmL water1	Standard	100	3.2	1331850.5
10_8_13 slixane QAQC 1000ngmLwater1	Standard	1000	3.19	6084538.0
10_8_13 slixane QAQC 10ngmLwater2	Standard	10	3.19	143883.8
10_8_13 slixane QAQC 100ngmLwater2	Standard	100	3.19	899188.1
10_8_13 slixane QAQC 1000ngmLwater2	Standard	1000	3.19	6170537.5
10_8_13 slixane QAQC 10ngmLwater3	Standard	10	3.19	104322.7

10_8_13 slixane QAQC 100ngmLwater3	Standard	100	3.19	988115.7
10_8_13 slixane QAQC 1000ngmLwater3	Standard	1000	3.19	6262216.5
10_8_13 slixane QAQC Blank2 sludge	Blank	0	3.2	56823.0
10_8_13 slixane QAQC 10ngmL sludge1	Analyte	10	3.2	107002.1
10_8_13 slixane QAQC 100ngmL sludge1	Analyte	100	3.2	433818.8
10_8_13 slixane QAQC 1000ngmL sludge1	Analyte	1000	3.2	4974870.0
10_8_13 slixane QAQC Blank3 sludge	Blank	0	3.2	51122.5
10_8_13 slixane QAQC 10ngmL sludge2	Analyte	10	3.2	104582.6
10_8_13 slixane QAQC 100ngmL sludge2	Analyte	100	3.2	490645.6
10_8_13 slixane QAQC 1000ngmL sludge2	Analyte	1000	3.2	4793780.0
10_8_13 slixane QAQC Blank4 sludge	Blank	0	3.2	47095.2
10_8_13 slixane QAQC 10ngmL sludge3	Analyte	10	3.2	97383.2
10_8_13 slixane QAQC 100ngmL sludge3	Analyte	100	3.2	366547.6
10_8_13 slixane QAQC 1000ngmL sludge3	Analyte	1000	3.2	4861868.5

Table B-18: Data of D5 QAQC analysis with split ratio of 1: 20 on 10/9/13

Name	Type	Std. Conc (ng·mL^{-1})	RT	Area
10_9_13 slixane QAQC Blank1	Blank	0	3.19	719.4
10_9_13 slixane QAQC 10ngmLwater1	Standard	10	3.19	24856.2
10_9_13 slixane QAQC 100ngmL water1	Standard	100	3.19	487978.0
10_9_13 slixane QAQC 1000ngmLwater1	Standard	1000	3.19	4188186.0
10_9_13 slixane QAQC 10ngmLwater2	Standard	10	3.19	35536.3
10_9_13 slixane QAQC 100ngmLwater2	Standard	100	3.19	341137.2
10_9_13 slixane QAQC 1000ngmLwater2	Standard	1000	3.19	3462726.3
10_9_13 slixane QAQC 10ngmLwater3	Standard	10	3.19	49880.5
10_9_13 slixane QAQC 100ngmLwater3	Standard	100	3.19	244939.4
10_9_13 slixane QAQC 1000ngmLwater3	Standard	1000	3.19	3299876.3
10_9_13 slixane QAQC 5ngmLwater	Analyte	5	3.19	18575.4
10_9_13 slixane QAQC 25ngmLwater	Analyte	25	3.19	82569.8
10_9_13 slixane QAQC 500ngmLwater	Analyte	500	3.19	1740548.1
10_9_13 slixane QAQC Blank2 sludge	Blank	0	3.2	16280.7
10_9_13 slixane QAQC 10ngmL sludge1	Analyte	10	3.19	43082.6
10_9_13 slixane QAQC 100ngmL sludge1	Analyte	100	3.2	440583.9
10_9_13 slixane QAQC 1000ngmL sludge1	Analyte	1000	3.19	2978801.8
10_9_13 slixane QAQC Blank3 sludge	Blank	0	3.19	15846.9
10_9_13 slixane QAQC 10ngmL sludge2	Analyte	10	3.19	35270.1
10_9_13 slixane QAQC 100ngmL sludge2	Analyte	100	3.19	251179.0
10_9_13 slixane QAQC 1000ngmL sludge2	Analyte	1000	3.19	2771061.8

10_9_13 slixane QAQC Blank4 sludge	Blank	0	3.19	15966.3
10_9_13 slixane QAQC 10ngmL sludge3	Analyte	10	3.19	36122.3
10_9_13 slixane QAQC 100ngmL sludge3	Analyte	100	3.19	273302.1
10_9_13 slixane QAQC 1000ngmL sludge3	Analyte	1000	3.19	2897739.5
10_9_13 slixane QAQC 5ngmL sludge	Analyte	5	3.2	33169.2
10_9_13 slixane QAQC 25ngmL sludge	Analyte	25	3.19	64275.1
10_9_13 slixane QAQC 500ngmL sludge	Analyte	500	3.19	1186211.6

Table B-19: Data of D5 QAQC analysis with split ratio of 1: 20 on 10/10/13

Name	Type	Std. Conc ($ng{\cdot}mL^{-1}$)	RT	Area
10_10_13 slixane QAQC Blank1	Blank	0	3.2	432.5
10_10_13 slixane QAQC 10ngmLwater1	Standard	10	3.2	22882.2
10_10_13 slixane QAQC 100ngmL water1	Standard	100	3.2	435063.8
10_10_13 slixane QAQC 1000ngmLwater1	Standard	1000	3.2	1939073.4
10_10_13 slixane QAQC 10ngmLwater2	Standard	10	3.2	34869.8
10_10_13 slixane QAQC 100ngmLwater2	Standard	100	3.2	146810.1
10_10_13 slixane QAQC 1000ngmLwater2	Standard	1000	3.2	2287376.3
10_10_13 slixane QAQC 10ngmLwater3	Standard	10	3.2	20397.6
10_10_13 slixane QAQC 100ngmLwater3	Standard	100	3.2	143599.0
10_10_13 slixane QAQC 1000ngmLwater3	Standard	1000	3.2	3965586.5
10_10_13 slixane QAQC 5ngmLwater	Analyte	5	3.2	16206.9
10_10_13 slixane QAQC 25ngmLwater	Analyte	25	3.2	53910.4
10_10_13 slixane QAQC 500ngmLwater	Analyte	500	3.2	1389830.5
10_10_13 slixane QAQC Blank2 sludge	Blank	0	3.21	13212.3
10_10_13 slixane QAQC 10ngmL sludge1	Standard	10	3.21	32161.1
10_10_13 slixane QAQC 100ngmL sludge1	Standard	100	3.21	241018.9
10_10_13 slixane QAQC 1000ngmL sludge1	Standard	1000	3.21	1535613.3
10_10_13 slixane QAQC Blank3 sludge	Blank	0	3.21	10332.1
10_10_13 slixane QAQC 10ngmL sludge2	Standard	10	3.21	30008.7
10_10_13 slixane QAQC 100ngmL sludge2	Standard	100	3.21	254013.3
10_10_13 slixane QAQC 1000ngmL sludge2	Standard	1000	3.21	2000647.6
10_10_13 slixane QAQC Blank4 sludge	Blank	0	3.21	14356.4
10_10_13 slixane QAQC 10ngmL sludge3	Standard	10	3.21	30147.6
10_10_13 slixane QAQC 100ngmL sludge3	Standard	100	3.21	96331.6
10_10_13 slixane QAQC 1000ngmL sludge3	Standard	1000	3.21	2509006.5
10_10_13 slixane QAQC 5ngmL sludge	Analyte	5	3.21	28249.8

10_10_13 slixane QAQC 25ngmL sludge	Analyte	25	3.21	85437.6
10_10_13 slixane QAQC 500ngmL sludge	Analyte	500	3.21	980588.1

Table B-20: Data of D5 during feasible test of split ratio on 11/1/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
11_1_2013 siloxanes sludge 0ngmL 1:5 split	Analyte		3.32	20379.3
11_1_2013 siloxanes water 50ngmL 1:5 split	Analyte	50	3.32	215.8
11_1_2013 siloxanes sludge 0ngmL 1:10 split	Analyte		3.23	100274.4
11_1_2013 siloxanes water 50ngmL 1:10 split	Analyte	50	3.23	10758.0

Table B-21: Data of D5 QAQC analysis with split ratio 1:10 on 11/2/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
11_2_2013 siloxanes water blank1	Blank		3.24	469.7
11_2_2013 siloxanes water 01ngmL 1	Standard	0.1	3.24	761.5
11_2_2013 siloxanes water 05ngmL 1	Standard	0.5	3.24	830.3
11_2_2013 siloxanes water 1 ngmL 1	Standard	1	3.24	1400.9
11_2_2013 siloxanes water 5 ngmL 1	Standard	5	3.24	9866.2
11_2_2013 siloxanes water 10 ngmL 1	Standard	10	3.24	5700.8
11_2_2013 siloxanes water 50 ngmL 1	Standard	50	3.24	82480.3
11_2_2013 siloxanes water blank 2	Blank		3.24	430.5
11_2_2013 siloxanes water 01ngmL 2	Standard	0.1	3.24	650.4
11_2_2013 siloxanes water 05ngmL 2	Standard	0.5	3.24	852.4
11_2_2013 siloxanes water 1 ngmL 2	Standard	1	3.24	1205.7
11_2_2013 siloxanes water 5 ngmL 2	Standard	5	3.24	8040.9
11_2_2013 siloxanes water 10 ngmL 2	Standard	10	3.24	7657.0
11_2_2013 siloxanes water 50 ngmL 2	Standard	50	3.24	68542.0
11_2_2013 siloxanes water blank 3	Blank		3.24	438.3
11_2_2013 siloxanes water 01ngmL 3	Standard	0.1	3.24	505.2
11_2_2013 siloxanes water 05ngmL 3	Standard	0.5	3.24	827.2
11_2_2013 siloxanes water 1 ngmL 3	Standard	1	3.24	1176.1
11_2_2013 siloxanes water 5 ngmL 3	Standard	5	3.24	4426.2
11_2_2013 siloxanes water 10 ngmL 3	Standard	10	3.24	7647.0
11_2_2013 siloxanes water 50 ngmL 3	Standard	50	3.24	40778.8
11_2_2013 siloxanes sludge blank1	Blank		3.24	8585.6
11_2_2013 siloxanes sludge 01ngmL 1	Analyte		3.25	7893.5
11_2_2013 siloxanes sludge 05ngmL 1	Analyte		3.25	8697.7
11_2_2013 siloxanes sludge 1 ngmL 1	Analyte		3.24	9101.2

11_2_2013 siloxanes sludge 5 ngmL 1	Analyte		3.25	10646.1
11_2_2013 siloxanes sludge 10 ngmL 1	Analyte		3.25	11293.5
11_2_2013 siloxanes sludge 50 ngmL 1	Analyte		3.24	24881.3
11_2_2013 siloxanes sludge blank 2	Blank		3.25	8849.3
11_2_2013 siloxanes sludge 01ngmL 2	Analyte		3.25	9904.8
11_2_2013 siloxanes sludge 05ngmL 2	Analyte		3.24	9915.5
11_2_2013 siloxanes sludge 1 ngmL 2	Analyte		3.25	8983.4
11_2_2013 siloxanes sludge 5 ngmL 2	Analyte		3.24	10855.9
11_2_2013 siloxanes sludge 10 ngmL 2	Analyte		3.24	13996.6
11_2_2013 siloxanes sludge 50 ngmL 2	Analyte		3.25	31143.8
11_2_2013 siloxanes sludge blank 3	Blank		3.25	8649.0
11_2_2013 siloxanes sludge 01ngmL 3	Analyte		3.24	10680.2
11_2_2013 siloxanes sludge 05ngmL 3	Analyte		3.25	9793.8
11_2_2013 siloxanes sludge 1 ngmL 3	Analyte		3.25	10594.4
11_2_2013 siloxanes sludge 5 ngmL 3	Analyte		3.24	13201.1
11_2_2013 siloxanes sludge 10 ngmL 3	Analyte		3.24	12350.2
11_2_2013 siloxanes sludge 50 ngmL 3	Analyte		3.24	29377.6

Table B-22: Data of D5 QAQC analysis with split ratio 1:10 on 11/3/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
11_3_2013 siloxanes water blank1	Blank		3.26	61.5
11_3_2013 siloxanes water 01ngmL 1	Standard	0.1	3.26	180.7
11_3_2013 siloxanes water 05ngmL 1	Standard	0.5	3.26	514.2
11_3_2013 siloxanes water 1 ngmL 1	Standard	1	3.26	985.5
11_3_2013 siloxanes water 5 ngmL 1	Standard	5	3.26	4108.9
11_3_2013 siloxanes water 10 ngmL 1	Standard	10	3.26	8466.1
11_3_2013 siloxanes water 50 ngmL 1	Standard	50	3.26	56368.0
11_3_2013 siloxanes water blank 2	Blank		3.26	92.0
11_3_2013 siloxanes water 01ngmL 2	Standard	0.1	3.26	192.5
11_3_2013 siloxanes water 05ngmL 2	Standard	0.5	3.26	502.1
11_3_2013 siloxanes water 1 ngmL 2	Standard	1	3.26	926.7
11_3_2013 siloxanes water 5 ngmL 2	Standard	5	3.26	4425.6
11_3_2013 siloxanes water 10 ngmL 2	Standard	10	3.26	7228.5
11_3_2013 siloxanes water 50 ngmL 2	Standard	50	3.26	50731.8
11_3_2013 siloxanes water blank 3	Blank		3.26	113.5
11_3_2013 siloxanes water 01ngmL 3	Standard	0.1	3.26	187.1
11_3_2013 siloxanes water 05ngmL 3	Standard	0.5	3.26	430.3
11_3_2013 siloxanes water 1 ngmL 3	Standard	1	3.26	729.8

11_3_2013 siloxanes water 5 ngmL 3	Standard	5	3.26	3357.9
11_3_2013 siloxanes water 10 ngmL 3	Standard	10	3.26	4806.0
11_3_2013 siloxanes water 50 ngmL 3	Standard	50	3.26	40184.7
11_3_2013 siloxanes sludge blank1	Blank		3.26	5946.3
11_3_2013 siloxanes sludge 01ngmL 1	Analyte		3.26	6928.3
11_3_2013 siloxanes sludge 05ngmL 1	Analyte		3.26	7039.1
11_3_2013 siloxanes sludge 1 ngmL 1	Analyte		3.25	7644.2
11_3_2013 siloxanes sludge 5 ngmL 1	Analyte		3.26	8851.4
11_3_2013 siloxanes sludge 10 ngmL 1	Analyte		3.26	9785.5
11_3_2013 siloxanes sludge 50 ngmL 1	Analyte		3.26	21974.3
11_3_2013 siloxanes sludge blank 2	Blank		3.26	7155.6
11_3_2013 siloxanes sludge 01ngmL 2	Analyte		3.26	7278.6
11_3_2013 siloxanes sludge 05ngmL 2	Analyte		3.25	7953.4
11_3_2013 siloxanes sludge 1 ngmL 2	Analyte		3.26	7590.0
11_3_2013 siloxanes sludge 5 ngmL 2	Analyte		3.26	8700.3
11_3_2013 siloxanes sludge 10 ngmL 2	Analyte		3.25	9491.6
11_3_2013 siloxanes sludge 50 ngmL 2	Analyte		3.26	25881.4
11_3_2013 siloxanes sludge blank 3	Blank		3.26	7346.9
11_3_2013 siloxanes sludge 01ngmL 3	Analyte		3.25	8728.3
11_3_2013 siloxanes sludge 05ngmL 3	Analyte		3.25	8068.4
11_3_2013 siloxanes sludge 1 ngmL 3	Analyte		3.25	8569.1
11_3_2013 siloxanes sludge 5 ngmL 3	Analyte		3.25	9344.8
11_3_2013 siloxanes sludge 10 ngmL 3	Analyte		3.25	10777.4
11_3_2013 siloxanes sludge 50 ngmL 3	Analyte		3.25	25373.8

Table B-23: Data of D5 QAQC analysis with split ratio 1:10 on 11/4/13

Name	Type	Std. Conc ($ng{\cdot}mL^{-1}$)	RT	Area
11_4_2013 siloxanes water blank1	Blank		3.24	48.6
11_4_2013 siloxanes water 01ngmL 1	Standard	0.1	3.24	248.3
11_4_2013 siloxanes water 05ngmL 1	Standard	0.5	3.24	490.9
11_4_2013 siloxanes water 1 ngmL 1	Standard	1	3.24	767.2
11_4_2013 siloxanes water 5 ngmL 1	Standard	5	3.24	4500.2
11_4_2013 siloxanes water 10 ngmL 1	Standard	10	3.24	6345.5
11_4_2013 siloxanes water 50 ngmL 1	Standard	50	3.24	51925.7
11_4_2013 siloxanes water blank 2	Blank		3.24	103.3
11_4_2013 siloxanes water 01ngmL 2	Standard	0.1	3.24	266.8
11_4_2013 siloxanes water 05ngmL 2	Standard	0.5	3.24	393.4
11_4_2013 siloxanes water 1 ngmL 2	Standard	1	3.24	804.0

11_4_2013 siloxanes water 5 ngmL 2	Standard	5	3.24	3319.2
11_4_2013 siloxanes water 10 ngmL 2	Standard	10	3.24	5433.9
11_4_2013 siloxanes water 50 ngmL 2	Standard	50	3.24	46586.8
11_4_2013 siloxanes water blank 3	Blank		3.24	128.5
11_4_2013 siloxanes water 01ngmL 3	Standard	0.1	3.24	203.5
11_4_2013 siloxanes water 05ngmL 3	Standard	0.5	3.24	448.2
11_4_2013 siloxanes water 1 ngmL 3	Standard	1	3.24	755.5
11_4_2013 siloxanes water 5 ngmL 3	Standard	5	3.24	4276.3
11_4_2013 siloxanes water 10 ngmL 3	Standard	10	3.24	5974.6
11_4_2013 siloxanes water 50 ngmL 3	Standard	50	3.24	48188.1
11_4_2013 siloxanes sludge blank1	Blank		3.24	5248.8
11_4_2013 siloxanes sludge 01ngmL 1	Analyte		3.24	5448.3
11_4_2013 siloxanes sludge 05ngmL 1	Analyte		3.24	5711.7
11_4_2013 siloxanes sludge 1 ngmL 1	Analyte		3.24	6666.9
11_4_2013 siloxanes sludge 5 ngmL 1	Analyte		3.24	6520.8
11_4_2013 siloxanes sludge 10 ngmL 1	Analyte		3.24	7744.8
11_4_2013 siloxanes sludge 50 ngmL 1	Analyte		3.24	15726.9
11_4_2013 siloxanes sludge blank 2	Blank		3.24	6174.5
11_4_2013 siloxanes sludge 01ngmL 2	Analyte		3.24	6213.6
11_4_2013 siloxanes sludge 05ngmL 2	Analyte		3.24	5304.0
11_4_2013 siloxanes sludge 1 ngmL 2	Analyte		3.24	6297.8
11_4_2013 siloxanes sludge 5 ngmL 2	Analyte		3.24	6755.7
11_4_2013 siloxanes sludge 10 ngmL 2	Analyte		3.24	8603.5
11_4_2013 siloxanes sludge 50 ngmL 2	Analyte		3.24	15613.8
11_4_2013 siloxanes sludge blank 3	Blank		3.24	5355.0
11_4_2013 siloxanes sludge 01ngmL 3	Analyte		3.24	6984.2
11_4_2013 siloxanes sludge 05ngmL 3	Analyte		3.24	6901.4
11_4_2013 siloxanes sludge 1 ngmL 3	Analyte		3.24	6999.1
11_4_2013 siloxanes sludge 5 ngmL 3	Analyte		3.24	8158.5
11_4_2013 siloxanes sludge 10 ngmL 3	Analyte		3.24	9284.8
11_4_2013 siloxanes sludge 50 ngmL 3	Analyte		3.24	15948.1

Table B-24: Data of D5 Fort Collins and Loveland analysis with split ratio 1:10 on 11/16/2013

Name	Type	Std. Conc	RT	Area
2013_11_16 Siloxanes water Blank 1	Water Blank	0	3.25	218.1
2013_11_16 Siloxanes water 0_1ngmL	Water Standard	0.5	3.25	608.2
2013_11_16 Siloxanes water 5 ngmL	Water Standard	5	3.25	5906.2
2013_11_16 Siloxanes water 50 ngmL	Water Standard	50	3.25	54548.4
2013_11_16 Siloxanes sludge Blank 2	Sludge Blank	0	3.25	7894.0

2013_11_16 Siloxanes sludge 0_1 ngmL	Sludge Standard	0.1	3.25	8583.3
2013_11_16 Siloxanes sludge 0_5 ngmL	Sludge Standard	0.5	3.25	9173.9
2013_11_16 Siloxanes sludge 1 ngmL	Sludge Standard	1	3.25	9545.6
2013_11_16 Siloxanes sludge 5 ngmL	Sludge Standard	5	3.25	11229.2
2013_11_16 Siloxanes sludge 10 ngmL	Sludge Standard	10	3.25	15549.9
2013_11_16 Siloxanes sludge 50 ngmL	Sludge Standard	50	3.25	50847.8
2013_11_16 Siloxanes water Blank 3	Blank		3.25	109.3
2013_11_16 Siloxanes FC 11 Effluent	Water Analyte		3.25	322.6
2013_11_16 Siloxanes FC 21 Influent	Water Analyte		3.25	860.1
2013_11_16 Siloxanes FC 31 1sludge	Sludge Analyte		3.25	11455.2
2013_11_16 Siloxanes FC 41 WAS	Sludge Analyte		3.25	12799.0
2013_11_16 Siloxanes FC 51 Digested sludge	Sludge Analyte		3.25	8165.8
2013_11_16 Siloxanes water Blank 4	Blank		3.25	95.5
2013_11_16 Siloxanes FC 12 Effluent	Water Analyte		3.25	273.2
2013_11_16 Siloxanes FC 22 Influent	Water Analyte		3.25	1222.3
2013_11_16 Siloxanes FC 32 1sludge	Sludge Analyte		3.25	10782.6
2013_11_16 Siloxanes FC 42 WAS	Sludge Analyte		3.25	11082.3
2013_11_16 Siloxanes FC 52 Digested sludge	Sludge Analyte		3.25	8041.2
2013_11_16 Siloxanes water Blank 5	Blank		3.26	99.5
2013_11_16 Siloxanes LL 11 Effluent	Water Analyte		3.25	328.7
2013_11_16 Siloxanes LL 21 PEF	Water Analyte		3.25	806.0
2013_11_16 Siloxanes LL 31 Primary Effluent	Water Analyte		3.25	858.2
2013_11_16 Siloxanes LL 41 Influent	Water Analyte		3.25	1072.0
2013_11_16 Siloxanes LL 51 2 digester	Sludge Analyte		3.25	4607.4
2013_11_16 Siloxanes LL 61 1 digester	Sludge Analyte		3.25	9547.6
2013_11_16 Siloxanes LL 71 Primary sludge	Sludge Analyte		3.25	8352.5
2013_11_16 Siloxanes LL 81 WAS	Sludge Analyte		3.25	1044.6
2013_11_16 Siloxanes water Blank 6	Blank		3.25	74.7
2013_11_16 Siloxanes LL 12 Effluent	Water Analyte		3.25	423.6
2013_11_16 Siloxanes LL 22 PEF	Water Analyte		3.25	1135.8
2013_11_16 Siloxanes LL 32 Primary Effluent	Water Analyte		3.25	1024.1
2013_11_16 Siloxanes LL 42 Influent	Water Analyte		3.25	1697.4
2013_11_16 Siloxanes LL 52 2 digester	Sludge Analyte		3.25	4279.3
2013_11_16 Siloxanes LL 62 1 digester	Sludge Analyte		3.25	10266.2

2013_11_16 Siloxanes LL 72 Primary sludge	Sludge Analyte		3.25	9841.7
2013_11_16 Siloxanes LL 82 WAS	Sludge Analyte		3.24	1129.4

Table B-25: Data of D5 Boulder analysis with split ratio 1:10 on 11/22/2013

Name	Type	Std. Conc	RT	Area
2013_11_22 Siloxanes water Blank 1	Blank		3.19	216.2
2013_11_22 Siloxanes water 0_1ngmL	Standard	0.5	3.19	524.0
2013_11_22 Siloxanes water 5 ngmL	Standard	5	3.19	6807.1
2013_11_22 Siloxanes water 50 ngmL	Standard	50	3.19	43061.4
2013_11_22 Siloxanes sludge Blank 2	Blank		3.19	9271.3
2013_11_22 Siloxanes sludge 0_1 ngmL	Standard	0.1	3.19	10071.7
2013_11_22Siloxanes sludge 0_5 ngmL	Standard	0.5	3.19	13682.7
2013_11_22 Siloxanes sludge 1 ngmL	Standard	1	3.19	14269.6
2013_11_22 Siloxanes sludge 5 ngmL	Standard	5	3.19	15020.5
2013_11_22 Siloxanes sludge 10 ngmL	Standard	10	3.19	14118.8
2013_11_22 Siloxanes sludge 50 ngmL	Standard	50	3.19	36207.8
2013_11_22 Siloxanes water Blank 3	Blank		3.19	181.2
2013_11_22 Siloxanes water Blank 4	Blank		3.19	205.1
2013_11_22 Siloxanes water Blank 5	Blank		3.2	155.3
2013_11_22 Siloxanes Rec 0_25ngmL1	Analyte		3.19	12770.2
2013_11_22 Siloxanes Rec 0_25ngmL2	Analyte		3.19	9175.2
2013_11_22 Siloxanes Rec 0_25ngmL3	Analyte		3.19	11757.2
2013_11_22 Siloxanes Rec 7_5 ngmL1	Analyte		3.18	19504.7
2013_11_22 Siloxanes Rec 7_5 ngmL2	Analyte		3.19	11975.9
2013_11_22 Siloxanes Rec 7_5 ngmL3	Analyte		3.19	14335.9
2013_11_22 Siloxanes Rec 25 ngmL 1	Analyte		3.19	26344.4
2013_11_22 Siloxanes Rec 25 ngmL 2	Analyte		3.19	27474.8
2013_11_22 Siloxanes Rec 25 ngmL 3	Analyte		3.19	26868.6
2013_11_22 Siloxanes BD Effluent 1	Analyte		3.19	150.9
2013_11_22 Siloxanes BD Effluent 2	Analyte		3.19	147.8
2013_11_22 Siloxanes BD Centrifuge 1	Analyte		3.19	797.2
2013_11_22 Siloxanes BD Centrifuge 2	Analyte		3.19	930.6
2013_11_22 Siloxanes BD Influent 1	Analyte		3.19	833.1
2013_11_22 Siloxanes BD Influent 2	Analyte		3.19	827.1
2013_11_22 Siloxanes BD Digester 1	Analyte		3.19	8096.9
2013_11_22 Siloxanes BD Digester 2	Analyte		3.19	9068.2
2013_11_22 Siloxanes BD WAS 1	Analyte		3.19	20178.8
2013_11_22 Siloxanes BD WAS 2	Analyte		3.19	18298.6

2013_11_22 Siloxanes Primary sludge 1	Analyte		3.19	15434.7
2013_11_22 Siloxanes Primary sludge 2	Analyte		3.19	17539.7

Table B-26: Data of D5 Fort Collins and Loveland analysis with split ratio 1:10 on 1/10/2014

Name	Type	Std. Conc	RT	Area
2014_1_10 siloxane blank1 water	Blank			
2014_1_10 siloxane 05D405D5water	Standard	0.5	3.22	1242.1
2014_1_10 siloxane 1D45D5 water	Standard	5	3.22	3201.5
2014_1_10 siloxane 5D450D5water	Standard	50	3.22	29577.7
2014_1_10 siloxane 15D4150D5 water	Standard	150	3.22	85402.4
2014_1_10 siloxane blank2 sludge	Blank		3.22	34064.6
2014_1_10 siloxane 05D405D5sludge	Standard	0.5	3.22	38568.6
2014_1_10 siloxane 1D45D5 sludge	Standard	5	3.22	28798.0
2014_1_10 siloxane 5D450D5sludge	Standard	50	3.22	43776.7
2014_1_10 siloxane 15D4150D5 sludge	Standard	150	3.22	55977.1
2014_1_10 siloxane blank3 water	Blank		3.22	775.7
2014_1_10 siloxane blank4 water	Blank		3.23	603.9
2014_1_10 siloxane blank5 water	Blank		3.22	677.4
2014_1_10 siloxane blank6 water	Blank		3.22	575.1
2014_1_10 siloxane FC effluent1	Analyte		3.22	752.0
2014_1_10 siloxane FC effluent2	Analyte		3.22	547.6
2014_1_10 siloxane FCinfluent1	Analyte		3.22	2657.3
2014_1_10 siloxane FCinfluent2	Analyte		3.22	2932.7
2014_1_10 siloxane FCcentrate1	Analyte		3.22	2002.2
2014_1_10 siloxane FCcentrate2	Analyte		3.22	1692.3
2014_1_10 siloxane FC primary sludge1	Analyte		3.23	22158.5
2014_1_10 siloxane FC primary sludge2	Analyte		3.22	26991.3
2014_1_10 siloxane FC WAS1	Analyte		3.22	69951.5
2014_1_10 siloxane FC WAS2	Analyte		3.22	64522.4
2014_1_10 siloxane FC RAS 1	Analyte		3.22	21406.7
2014_1_10 siloxane FC RAS 2	Analyte		3.22	23726.3
2014_1_10 siloxane FC anaerobic sludge1	Analyte		3.22	24795.5
2014_1_10 siloxane FC anaerobic sludge2	Analyte		3.22	28007.7
2014_1_10 siloxane LL effluent 1	Analyte		3.22	669.1
2014_1_10 siloxane LL effluent 2	Analyte		3.22	896.4
2014_1_10 siloxane LL primary effluent 1	Analyte		3.22	2038.3
2014_1_10 siloxane LL primary effluent 2	Analyte		3.22	2188.8

2014_1_10 siloxane LL influent 1	Analyte		3.22	1609.5
2014_1_10 siloxane LL influent 2	Analyte		3.22	1788.8
2014_1_10 siloxane LL Centrate 1	Analyte		3.22	2301.6
2014_1_10 siloxane LL Centrate 2	Analyte		3.22	2683.2
2014_1_10 siloxane LL primary digester 1	Analyte		3.22	20478.1
2014_1_10 siloxane LL primary digester 2	Analyte		3.22	23776.2
2014_1_10 siloxane LL RAS 1	Analyte		3.22	6045.5
2014_1_10 siloxane LL RAS 2	Analyte		3.22	5867.3
2014_1_10 siloxane LL Primary sludge 1	Analyte		3.22	8810.9
2014_1_10 siloxane LL Primary sludge 2	Analyte		3.22	8435.1

Table B-27: Data of D5 Fort Collins and Loveland analysis with split ratio 1:10 on 2/5/2014

Name	Type	Std. Conc	RT	Area
2014_2_5 siloxane blank1 water	Blank		3.2	547.2
2014_2_5 siloxane 05D405D5water	Standard	0.5	3.2	951.0
2014_2_5 siloxane 1D45D5 water	Standard	5	3.2	2839.1
2014_2_5 siloxane 5D450D5water	Standard	50	3.2	28891.3
2014_2_5 siloxane 15D4150D5 water	Standard	150	3.2	81358.6
2014_2_5 siloxane blank2 sludge	Blank		3.19	23781.1
2014_2_5 siloxane 05D405D5sludge	Standard	0.5	3.19	24945.4
2014_2_5 siloxane 1D45D5 sludge	Standard	5	3.2	26007.4
2014_2_5 siloxane 5D450D5sludge	Standard	50	3.19	37262.7
2014_2_5 siloxane 15D4150D5 sludge	Standard	150	3.2	67002.4
2014_2_5 siloxane blank3 water	Blank		3.2	613.0
2014_2_5 siloxane blank4 water	Blank		3.2	648.5
2014_2_6 siloxanes Blanck water 5	Blank		3.21	13889.5
2014_2_5 siloxane Blank sludge	Blank		3.2	11822.6
2014_2_5 siloxane LL effluent 1	Analyte		3.2	740.7
2014_2_5 siloxane LL effluent 2	Analyte		3.2	925.2
2014_2_5 siloxane LL primary effluent 1	Analyte		3.2	1869.3
2014_2_5 siloxane LL primary effluent 2	Analyte		3.2	1827.8
2014_2_5 siloxane LL influent 1	Analyte		3.2	1365.2
2014_2_5 siloxane LL influent 2	Analyte		3.2	1361.6
2014_2_5 siloxane LL Centrate 1	Analyte		3.2	9806.3
2014_2_5 siloxane LL Centrate 2	Analyte		3.2	11113.3
2014_2_5 siloxane LL primary digester 1	Analyte		3.2	10999.0
2014_2_5 siloxane LL primary digester 2	Analyte		3.2	11843.9
2014_2_5 siloxane LL primary sludge 1	Analyte		3.2	20327.0
2014_2_5 siloxane LL Primary sludge 2	Analyte		3.2	20770.9

2014_2_5 siloxane LL secondary sludge 1	Analyte		3.2	4379.7
2014_2_5 siloxane LL secondary sludge2	Analyte		3.19	3759.9
2014_2_5 siloxane LL Primary clarifier 1	Analyte		3.2	1558.0
2014_2_5 siloxane LL Primary clarifier 2	Analyte		3.19	2058.5
2014_2_6 siloxanes FC eff 1	Analyte		3.21	849.8
2014_2_6 siloxanes FC eff 2	Analyte		3.21	961.0
2014_2_6 siloxanes FC inf 1	Analyte		3.21	1235.8
2014_2_6 siloxanes FC inf 2	Analyte		3.21	1411.3
2014_2_6 siloxanes FC Cen 1	Analyte		3.21	1155.3
2014_2_6 siloxanes FC Cen 2	Analyte		3.21	1165.8
2014_2_6 siloxanes FC Pri sludge 1	Analyte		3.21	15026.4
2014_2_6 siloxanes FC Pri sludge 2	Analyte		3.22	15718.5
2014_2_6 siloxanes FC TWAS 1	Analyte		3.21	27870.0
2014_2_6 siloxanes FC TWAS 2	Analyte		3.21	27958.1
2014_2_6 siloxanes FC RAS 1	Analyte		3.21	18947.1
2014_2_6 siloxanes FC RAS 2	Analyte		3.21	19553.4
2014_2_6 siloxanes FC Anaerobic Sludge 1	Analyte		3.21	14519.7
2014_2_6 siloxanes FC Anaerobic Sludge 2	Analyte		3.22	14489.0

Table B-28: Data of D5 Fort Collins and Loveland analysis with split ratio 1:10 on 3/5/2014

Name	Type	Std. Conc	RT	Area
2014_3_5 siloxanes Blank 1	Blank	0	3.2	337.1
2014_3_5 siloxanes water 05D405D5	Standard	0.5	3.2	577.4
2014_3_5 siloxanes water 1D45D5	Standard	5	3.2	2465.4
2014_3_5 siloxanes water 5D450D5	Standard	50	3.2	20411.0
2014_3_5 siloxanes water15D4150D5	Standard	150	3.2	59658.2
2014_3_5 siloxanes sludge Blank 2	Blank	0	3.2	15203.1
2014_3_5 siloxanes sludge 05D405D5	Standard	0.5	3.2	15943.0
2014_3_5 siloxanes sludge 1D45D5	Standard	5	3.2	18222.2
2014_3_5 siloxanes sludge 5D450D5	Standard	50	3.2	23118.8
2014_3_5 siloxanes sludge15D4150D5	Standard	150	3.2	36251.4
2014_3_5 siloxanes water Blank 3	Blank	0	3.21	221.2
2014_3_5 siloxanes water Blank 4	Blank	0	3.21	143.8
2014_3_6 siloxanes FC water blank1	Blank		3.21	79.1
2014_3_6 siloxanes FC water blank 2	Blank		3.21	297.4
2014_3_5 siloxanes LL effluent1	Analyte		3.2	225.0
2014_3_5 siloxanes LL effluent 2	Analyte		3.2	193.9
2014_3_5 siloxanes LL PEF 1	Analyte		3.2	722.8
2014_3_5 siloxanes LL PEF 2	Analyte		3.2	673.4

2014_3_5 siloxanes LL Influent 1	Analyte		3.2	685.9
2014_3_5 siloxanes LL Influent 2	Analyte		3.2	605.9
2014_3_5 siloxanes LL RAS 1	Analyte		3.2	1769.4
2014_3_5 siloxanes LL RAS 2	Analyte		3.2	1942.7
2014_3_5 siloxanes LL Primary Digester 1	Analyte		3.2	6893.8
2014_3_5 siloxanes LL Primary Digester 2	Analyte		3.2	6205.6
2014_3_5 siloxanes LL Primary sludge 1	Analyte		3.2	8895.9
2014_3_5 siloxanes LL Primary sludge 2	Analyte		3.2	7601.3
2014_3_6 siloxanes FC effluent 1	Analyte		3.21	185.5
2014_3_6 siloxanes FC effluent 2	Analyte		3.21	173.1
2014_3_6 siloxanes FC influent 1	Analyte		3.21	460.3
2014_3_6 siloxanes FC influent 2	Analyte		3.21	453.5
2014_3_6 siloxanes FC centrate 1	Analyte		3.21	768.6
2014_3_6 siloxanes FC centrate 2	Analyte		3.21	540.0
2014_3_6 siloxanes FC Primary sludge 1	Analyte		3.21	9721.9
2014_3_6 siloxanes FC Primary sludge 2	Analyte		3.21	10912.8
2014_3_6 siloxanes FC TWAS 1	Analyte		3.21	9679.5
2014_3_6 siloxanes FC TWAS 2	Analyte		3.21	8745.4
2014_3_6 siloxanes FC RAS 1	Analyte		3.21	7054.8
2014_3_6 siloxanes FC RAS 2	Analyte		3.22	5913.9
2014_3_6 siloxanes FC Anaerobic sludge 1	Analyte		3.21	6344.1
2014_3_6 siloxanes FC Anaerobic sludge 2	Analyte		3.21	6627.0

Table B-29: Data of D5 Fort Collins and Loveland analysis with split ratio 1:10 on 4/16/2014

Name	Type	Std. Conc	RT	Area
2014_4_16 siloxanes water 05D405D5	Standard	0.5	3.19	242.361
2014_4_16 siloxanes water 1D45D5	Standard	5	3.19	385.385
2014_4_16 siloxanes water 5D450D5	Standard	50	3.19	7895.813
2014_4_16 siloxanes water 15D4150D5	Standard	150	3.19	27150.26
2014_4_16 siloxanes sludge 05D405D5	Standard	0.5	3.19	4263.634
2014_4_16 siloxanes sludge 1D45D5	Standard	5	3.19	5045.715
2014_4_16 siloxanes sludge 5D450D5	Standard	50	3.19	7999.664
2014_4_16 siloxanes sludge 15D4150D5	Standard	150	3.19	14218.13
2014_4_16 siloxanes blank water 1	Blank		3.19	202.745
2014_4_16 silxoanes blank sludge 2	Blank		3.19	3887.344
2014_4_16 siloxanes Water blank 3	Blank		3.18	137.078
2014_4_16 siloxanes Water blank 4	Blank		3.19	108.229
2014_4_16 siloxanes water blank 5	Blank		3.18	122.18
2014_4_16 siloxanes water blank 6	Blank		3.19	84.529
2014_4_16 siloxanes FC effluent 1	Analyte		3.19	212.79

2014_4_16 siloxanes FC effluent 2	Analyte		3.18	187.778
2014_4_16 siloxanes FC Influent 1	Analyte		3.19	449.072
2014_4_16 siloxanes FC Influent 2	Analyte		3.19	389.958
2014_4_16 siloxanes FC Centrate 1	Analyte		3.19	231.472
2014_4_16 siloxanes FC Centrate 2	Analyte		3.19	311.015
2014_4_16 siloxanes FC Primary sludge 1	Analyte		3.19	5878.263
2014_4_16 siloxanes FC Primary sludge 2	Analyte		3.19	6397.505
2014_4_16 siloxanes FC TWAS 1	Analyte		3.19	8546.214
2014_4_16 siloxanes FC TWAS 2	Analyte		3.19	8912.3
2014_4_16 siloxanes FC RAS 1	Analyte		3.19	6257.282
2014_4_16 siloxanes FC RAS 2	Analyte		3.19	6936.946
2014_4_16 siloxanes FC Anaerobic sludge 1	Analyte		3.19	5174.136
2014_4_16 siloxanes FC Anaerobic sludge 2	Analyte		3.19	4731.021
2014_4_16 siloxanes LL effluent 1	Analyte		3.18	158.735
2014_4_16 siloxanes LL effluent 2	Analyte		3.19	132.369
2014_4_16 siloxanes LL primary effluent 1	Analyte		3.18	524.14
2014_4_16 siloxanes LL primary effluent 2	Analyte		3.19	448.29
2014_4_16 siloxanes LL influent 1	Analyte		3.19	484.511
2014_4_16 siloxanes LL influent 2	Analyte		3.19	565.904
2014_4_16 siloxanes LL Centrate 1	Analyte		3.19	54.318
2014_4_16 siloxanes LL Centrate 2	Analyte		3.19	60.476
2014_4_16 siloxanes LL RAS1	Analyte		3.19	2949.343
2014_4_16 siloxanes LL RAS 2	Analyte		3.19	2266.628
2014_4_16 siloxanes LL Primary digestion 1	Analyte		3.19	3483.028
2014_4_16 siloxanes LL Primary digestion 2	Analyte		3.19	3614.333
2014_4_16 siloxanes LL primary sludge 1	Analyte		3.19	2868.825
2014_4_16 siloxanes LL primary sludge 2	Analyte		3.19	3445.887

Table B-30: Data of D5 Fort Collins and Loveland analysis with split ratio 1:10 on 5/20/2014

Name	Type	Std. Conc	RT	Area
2014_5_20 siloxane blank water 1	Blank		3.17	384.738
2014_5_20 siloxane water 0505ngmL	Standard	0.5	3.16	629.553
2014_5_20 siloxane water D41D55ngmL	Standard	1	3.17	5653.848
2014_5_20 siloxane water D45D550ngmL	Standard	5	3.17	37373.95
2014_5_20 siloxane water D415D5150ngmL	Standard	15	3.17	115060.1
2014_5_21 siloxanes sludge blank	Blank		3.15	19979.51
2014_5_20 siloxane sludge D405D505ngmL	Standard	0.5	3.16	29127.89
2014_5_20 siloxane sludge D41D55ngmL	Standard	1	3.16	29719.1
2014_5_20 siloxane sludge D45D550ngmL	Standard	5	3.16	40528.24
2014_5_20 siloxane sludge D415D5150ngmL	Standard	15	3.16	62715.11
2014_5_20 siloxane blank water 00	Blank		3.15	268.212

2014_5_20 siloxane blank water 2	Blank		3.17	259.569
2014_5_20 siloxane blank water 3	Blank		3.16	163.276
2014_5_20 siloxane blank water 4	Blank		3.17	205.513
2014_5_20 siloxane blank water 5	Blank		3.17	206.066
2014_5_20 siloxane FC Effluent 1	Analyte		3.17	633.64
2014_5_20 siloxane FC Effluent 2	Analyte		3.17	655.92
2014_5_20 siloxane FC influent 1	Analyte		3.17	1527.793
2014_5_20 siloxane FC influent 2	Analyte		3.16	1414.722
2014_5_20 siloxane FC centrate 1	Analyte		3.16	787.835
2014_5_20 siloxane FC centrate 2	Analyte		3.16	803.674
2014_5_20 siloxane FC primary sludge 1	Analyte		3.16	26108.49
2014_5_20 siloxane FC primary sludge 2	Analyte		3.16	30746.6
2014_5_20 siloxane FC TWAS 1	Analyte		3.16	34065.75
2014_5_20 siloxane FC TWAS 2	Analyte		3.16	32286.79
2014_5_20 siloxane FC RAS 1	Analyte		3.16	17020.83
2014_5_20 siloxane FC RAS 2	Analyte		3.16	17078.6
2014_5_20 siloxane FC Digester 1	Analyte		3.16	16948.09
2014_5_20 siloxane FC Digester 2	Analyte		3.16	22153.79
2014_5_20 siloxane LL Effluent 1	Analyte		3.17	363.458
2014_5_20 siloxane LL Effluent 2	Analyte		3.16	375.197
2014_5_20 siloxane LL Primary effluent 1	Analyte		3.17	1864.44
2014_5_20 siloxane LL Primary effluent 2	Analyte		3.17	1895.981
2014_5_20 siloxane LL influent 1	Analyte		3.17	1398.619
2014_5_20 siloxane LL influent 2	Analyte		3.16	1353.147
2014_5_20 siloxane LL Centrate 1	Analyte		3.17	1765.026
2014_5_20 siloxane LL Centrate 2	Analyte		3.17	1924.583
2014_5_20 siloxane LL RAS 1	Analyte		3.15	41838.25
2014_5_20 siloxane LL RAS 2	Analyte		3.15	41893.93
2014_5_20 siloxane LL Digester 1	Analyte		3.15	18877.47
2014_5_20 siloxane LL Digester 2	Analyte		3.15	22672.65
2014_5_20 siloxane LL Primary sludge 1	Analyte		3.15	34603.43
2014_5_20 siloxane LL Primary sludge 2	Analyte		3.15	26771.66

Table B-31: Data of D5 Volume of Acetone Impact on 5/20/2014

Name	Type	Std. Conc	RT	Area
2014_5_20 siloxane acetone 50uL water	Analyte		3.16	3506.156
2014_5_20 siloxane acetone 100uL water	Analyte		3.15	6218.016
2014_5_20 siloxane acetone 500uL water	Analyte		3.12	13265.81
2014_5_20 siloxane acetone 1000uL water	Analyte		3.14	17520.59
2014_5_20 siloxane acetone 1500uL water	Analyte		3.11	14283.06
2014_5_20 siloxane acetone 1500uL water2	Analyte		3.12	8857.001

2014_5_20 siloxane acetone 50uL sludge	Analyte		3.16	34439.53
2014_5_20 siloxane acetone 100uL sludge	Analyte		3.16	32594.69
2014_5_20 siloxane acetone 500uL sludge	Analyte		3.14	73684.69
2014_5_20 siloxane acetone 1000uL sludge	Analyte		3.14	142248.1
2014_5_20 siloxane acetone 1500uL sludge	Analyte		3.12	188726.9
2014_5_20 siloxane acetone 1500uL sludge2	Analyte		3.13	164685.6

C. Raw Data – M4Q

Table C-1: Data of M4Q analysis on 9/4/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_4_13 Siloxane_Blank1	Blank	10	2.43	210365.8
9_4_13 Siloxane_1ngml	Standard	10	2.43	62082.4
9_4_13 Siloxane_2_5ngml	Standard	10	2.43	97610.8
9_4_13 Siloxane_7_5ngml	Standard	10	2.43	20548.4
9_4_13 Siloxane_Blank2	Blank	10	2.43	121253.9
9_4_13 Siloxane_sludge1	Analyte	10	2.44	81305.4
9_4_13 Siloxane_sludge2	Analyte	10	2.43	79602.3
9_4_13 Siloxane_Blank3	Blank	10	2.43	128761.9

Table C-2: Data of M4Q analysis on 9/10/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_10_13 Siloxane	Blank			
9_10_13 Siloxane_blank1	Blank		2.43	9138.4
9_10_13 Siloxane1ngml	Standard	10	2.43	4561.3
9_10_13 Siloxane2_5ngml	Standard	10	2.42	3316.0

Table C-3: Data of M4Q during blank test on 9/11/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_11_2013 Acetone	Blank			
9_11_2013 Water	Blank		2.78	0.0
9_11_2013 IS	Analyte		2.42	3915.4

Table C-4: Data of M4Q analysis repeated on 9/11/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_11_2013 Siloxaneblank	Blank	0	2.42	4199.0
9_11_2013 Siloxane1ngml	Standard	0.1	2.43	1930.7
9_11_2013 Siloxane2_5ngml	Standard	0.1	2.42	2932.5
9_11_2013 Siloxane7_5ngml	Standard	0.1	2.42	3005.1

Table C-5: Data of M4Q analysis on 9/12/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
9_12_2013 Siloxaneblank	Blank	0.01	2.41	203.4
9_12_2013 Siloxane1ngml	Standard	0.01	2.41	394.4
9_12_2013 Siloxane2_5ngml	Standard	0.01	2.41	200.8
9_12_2013 Siloxane7_5ngml	Standard	0.01	2.42	400.6

Table C-6: Data of M4Q in water analysis on 9/30/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
2013_9_30Siloxans_water 1	Standard	0.01	2.42	159.8
2013_9_30Siloxans_water 2	Standard	0.01	2.42	1091.2
2013_9_30Siloxans_water 3	Standard	0.01	2.42	2233.8

Table C-7: Data of M4Q in sludge analysis on 9/30/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
2013_9_30Siloxans_sludge 1	Blank	0.01	2.41	10.1
2013_9_30Siloxans_sludge 2	Standard	0.01	2.42	58.6
2013_9_30Siloxans_sludge 3	Standard	0.01	2.42	220.8
2013_9_30Siloxans_sludge 4	Standard	0.01	2.42	716.1

Table C-8: Data of M4Q analysis on 10/1/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
2013_10_1 Siloxane 0ngmL	Blank	0.01	2.43	1867986.6
2013_10_1 Siloxane 10ngmL	Analyte	0.01	2.44	1828879.8

Table C-9: Data of M4Q analysis with initial temperature of 60 ℃ on 10/4/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_4_2013 Siloxanes IS 60 C	Blank	0.01	3.71	1270101.4

Table C-10: Data of M4Q analysis with initial temperature of 70 ℃ on 10/4/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
10_4_2013 Siloxane full scan	Analyte	0.01		
10_4_2013 Siloxane IS with 70C	Analyte	0.01	3.3	1768744.5

Table C-11: Data of M4Q analysis with initial temperature of 80 ℃ on 10/7/13

Name	Type	Std. Conc (ng·mL^{-1})	RT	Area
10_7_13 Siloxane at 80C		0.5	3.01	365038.9

Table C-12: Data of M4Q analysis with initial temperature of 75 ℃ on 10/7/13

Name	Type	Std. Conc (ng·mL^{-1})	RT	Area
10_7_13 Siloxane at 75C			3.33	435856.0

Table C-13: Data of M4Q QAQC analysis with split ratio of 1: 20 on 10/8/13

Name	Type	Std. Conc (ng·mL^{-1})	RT	Area
10_8_13 slixane QAQC Blank1	Blank	0.5	3.33	2629662.8
10_8_13 slixane QAQC 10ngmLwater1	Standard	0.5	3.34	2375763.0
10_8_13 slixane QAQC 100ngmL water1	Standard	0.5	3.34	2853165.3
10_8_13 slixane QAQC 1000ngmLwater1	Standard	0.5	3.34	3188363.0
10_8_13 slixane QAQC 10ngmLwater2	Standard	0.5	3.33	3400358.3
10_8_13 slixane QAQC 100ngmLwater2	Standard	0.5	3.34	1123686.8
10_8_13 slixane QAQC 1000ngmLwater2	Standard	0.5	3.34	2221236.5
10_8_13 slixane QAQC 10ngmLwater3	Standard	0.5	3.33	2183554.5
10_8_13 slixane QAQC 100ngmLwater3	Standard	0.5	3.34	2289452.8
10_8_13 slixane QAQC 1000ngmLwater3	Standard	0.5	3.34	2107993.8
10_8_13 slixane QAQC Blank2 sludge	Blank	0.5	3.34	775825.4
10_8_13 slixane QAQC 10ngmL sludge1	Analyte	0.5	3.34	835224.6
10_8_13 slixane QAQC 100ngmL sludge1	Analyte	0.5	3.34	410586.7
10_8_13 slixane QAQC 1000ngmL sludge1	Analyte	0.5	3.34	955961.9
10_8_13 slixane QAQC Blank3 sludge	Blank	0.5	3.34	976644.1
10_8_13 slixane QAQC 10ngmL sludge2	Analyte	0.5	3.34	1064957.5
10_8_13 slixane QAQC 100ngmL sludge2	Analyte	0.5	3.34	825200.1
10_8_13 slixane QAQC 1000ngmL sludge2	Analyte	0.5	3.35	1110505.6
10_8_13 slixane QAQC Blank4 sludge	Blank	0.5	3.34	655010.2
10_8_13 slixane QAQC 10ngmL sludge3	Analyte	0.5	3.34	922173.3
10_8_13 slixane QAQC 100ngmL sludge3	Analyte	0.5	3.34	431775.3
10_8_13 slixane QAQC 1000ngmL sludge3	Analyte	0.5	3.34	962038.9

Table C-14: Data of M4Q QAQC analysis with split ratio of 1: 20 on 10/9/13

Name	Type	Std. Conc ($ng\cdot mL^{-1}$)	RT	Area
10_9_13 slixane QAQC Blank1	Blank	0.5	3.33	1738580.8
10_9_13 slixane QAQC 10ngmLwater1	Standard	0.5	3.33	407426.3
10_9_13 slixane QAQC 100ngmL water1	Standard	0.5	3.33	944088.3
10_9_13 slixane QAQC 1000ngmLwater1	Standard	0.5	3.33	626001.9
10_9_13 slixane QAQC 10ngmLwater2	Standard	0.5	3.33	706188.9
10_9_13 slixane QAQC 100ngmLwater2	Standard	0.5	3.33	1398878.6
10_9_13 slixane QAQC 1000ngmLwater2	Standard	0.5	3.33	472546.2
10_9_13 slixane QAQC 10ngmLwater3	Standard	0.5	3.33	270496.3
10_9_13 slixane QAQC 100ngmLwater3	Standard	0.5	3.33	1318462.1
10_9_13 slixane QAQC 1000ngmLwater3	Standard	0.5	3.33	382306.1
10_9_13 slixane QAQC 5ngmLwater	Analyte	0.5	3.33	363752.5
10_9_13 slixane QAQC 25ngmLwater	Analyte	0.5	3.33	164769.1
10_9_13 slixane QAQC 500ngmLwater	Analyte	0.5	3.33	247868.2
10_9_13 slixane QAQC Blank2 sludge	Blank	0.5	3.34	751850.3
10_9_13 slixane QAQC 10ngmL sludge1	Analyte	0.5	3.34	377021.5
10_9_13 slixane QAQC 100ngmL sludge1	Analyte	0.5	3.34	375880.8
10_9_13 slixane QAQC 1000ngmL sludge1	Analyte	0.5	3.34	379339.2
10_9_13 slixane QAQC Blank3 sludge	Blank	0.5	3.34	664869.3
10_9_13 slixane QAQC 10ngmL sludge2	Analyte	0.5	3.34	359983.7
10_9_13 slixane QAQC 100ngmL sludge2	Analyte	0.5	3.34	420330.3
10_9_13 slixane QAQC 1000ngmL sludge2	Analyte	0.5	3.34	439864.5
10_9_13 slixane QAQC Blank4 sludge	Blank	0.5	3.34	397677.9
10_9_13 slixane QAQC 10ngmL sludge3	Analyte	0.5	3.34	650349.0
10_9_13 slixane QAQC 100ngmL sludge3	Analyte	0.5	3.34	598186.8
10_9_13 slixane QAQC 1000ngmL sludge3	Analyte	0.5	3.33	207136.4
10_9_13 slixane QAQC 5ngmL sludge	Analyte	0.5	3.34	210508.0
10_9_13 slixane QAQC 25ngmL sludge	Analyte	0.5	3.34	220351.4
10_9_13 slixane QAQC 500ngmL sludge	Analyte	0.5	3.33	164847.2

Table C-15: Data of M4Q QAQC analysis with split ratio of 1: 20 on 10/10/13

Name	Type	Std. Conc ($ng\cdot mL^{-1}$)	RT	Area
10_10_13 slixane QAQC Blank1	Blank	0.5	3.35	2454851.0
10_10_13 slixane QAQC 10ngmLwater1	Standard	0.5	3.35	1649822.8
10_10_13 slixane QAQC 100ngmL water1	Standard	0.5	3.35	3773431.5
10_10_13 slixane QAQC 1000ngmLwater1	Standard	0.5	3.35	1851143.1

10_10_13 slixane QAQC 10ngmLwater2	Standard	0.5	3.35	4101076.3
10_10_13 slixane QAQC 100ngmLwater2	Standard	0.5	3.35	3127185.5
10_10_13 slixane QAQC 1000ngmLwater2	Standard	0.5	3.35	1426020.8
10_10_13 slixane QAQC 10ngmLwater3	Standard	0.5	3.35	1501693.1
10_10_13 slixane QAQC 100ngmLwater3	Standard	0.5	3.35	1017229.6
10_10_13 slixane QAQC 1000ngmLwater3	Standard	0.5	3.35	760008.2
10_10_13 slixane QAQC 5ngmLwater	Analyte	0.5	3.35	1147632.6
10_10_13 slixane QAQC 25ngmLwater	Analyte	0.5	3.35	804928.4
10_10_13 slixane QAQC 500ngmLwater	Analyte	0.5	3.35	1515786.8
10_10_13 slixane QAQC Blank2 sludge	Blank	0.5	3.36	500799.2
10_10_13 slixane QAQC 10ngmL sludge1	Standard	0.5	3.36	1015007.3
10_10_13 slixane QAQC 100ngmL sludge1	Standard	0.5	3.36	1710191.3
10_10_13 slixane QAQC 1000ngmL sludge1	Standard	0.5	3.36	962151.9
10_10_13 slixane QAQC Blank3 sludge	Blank	0.5	3.36	798070.4
10_10_13 slixane QAQC 10ngmL sludge2	Standard	0.5	3.36	809649.6
10_10_13 slixane QAQC 100ngmL sludge2	Standard	0.5	3.36	1678163.4
10_10_13 slixane QAQC 1000ngmL sludge2	Standard	0.5	3.36	918343.7
10_10_13 slixane QAQC Blank4 sludge	Blank	0.5	3.36	374160.0
10_10_13 slixane QAQC 10ngmL sludge3	Standard	0.5	3.36	990635.1
10_10_13 slixane QAQC 100ngmL sludge3	Standard	0.5	3.36	630012.6
10_10_13 slixane QAQC 1000ngmL sludge3	Standard	0.5	3.36	1160940.9
10_10_13 slixane QAQC 5ngmL sludge	Analyte	0.5	3.36	919659.9
10_10_13 slixane QAQC 25ngmL sludge	Analyte	0.5	3.36	776898.6
10_10_13 slixane QAQC 500ngmL sludge	Analyte	0.5	3.36	539659.6

Table C-16: Data of M4Q during feasible test of split ratio on 11/1/13

Name	Type	Std. Conc (ng·mL^{-1})	RT	Area
11_1_2013 siloxanes sludge 0ngmL 1:5 split	Analyte	0.1	3.47	4155.7
11_1_2013 siloxanes water 50ngmL 1:5 split	Analyte	0.1	3.54	3755.3
11_1_2013 siloxanes sludge 0ngmL 1:10 split	Analyte	0.1	3.38	22480.6
11_1_2013 siloxanes water 50ngmL 1:10 split	Analyte	0.1	3.38	68293.1

Table C-17: Data of M4Q QAQC analysis with split ratio 1:10 on 11/2/13

Name	Type	Std. Conc (ng·mL^{-1})	RT	Area
11_2_2013 siloxanes water blank1	Blank	0.1	3.38	11042.0
11_2_2013 siloxanes water 01ngmL 1	Standard	0.1	3.38	13984.7
11_2_2013 siloxanes water 05ngmL 1	Standard	0.1	3.38	13769.7

11_2_2013 siloxanes water 1 ngmL 1	Standard	0.1	3.38	13834.9
11_2_2013 siloxanes water 5 ngmL 1	Standard	0.1	3.38	14363.9
11_2_2013 siloxanes water 10 ngmL 1	Standard	0.1	3.38	13311.6
11_2_2013 siloxanes water 50 ngmL 1	Standard	0.1	3.38	13487.0
11_2_2013 siloxanes water blank 2	Blank	0.1	3.38	12902.7
11_2_2013 siloxanes water 01ngmL 2	Standard	0.1	3.38	12728.0
11_2_2013 siloxanes water 05ngmL 2	Standard	0.1	3.38	12644.3
11_2_2013 siloxanes water 1 ngmL 2	Standard	0.1	3.38	13254.6
11_2_2013 siloxanes water 5 ngmL 2	Standard	0.1	3.38	13101.7
11_2_2013 siloxanes water 10 ngmL 2	Standard	0.1	3.38	13710.9
11_2_2013 siloxanes water 50 ngmL 2	Standard	0.1	3.38	13475.4
11_2_2013 siloxanes water blank 3	Blank	0.1	3.38	11966.3
11_2_2013 siloxanes water 01ngmL 3	Standard	0.1	3.38	12904.2
11_2_2013 siloxanes water 05ngmL 3	Standard	0.1	3.38	12435.8
11_2_2013 siloxanes water 1 ngmL 3	Standard	0.1	3.39	12026.2
11_2_2013 siloxanes water 5 ngmL 3	Standard	0.1	3.38	12704.7
11_2_2013 siloxanes water 10 ngmL 3	Standard	0.1	3.38	12141.2
11_2_2013 siloxanes water 50 ngmL 3	Standard	0.1	3.39	11554.3
11_2_2013 siloxanes sludge blank1	Blank	0.1	3.38	45312.2
11_2_2013 siloxanes sludge 01ngmL 1	Analyte	0.1	3.38	57043.6
11_2_2013 siloxanes sludge 05ngmL 1	Analyte	0.1	3.38	57361.3
11_2_2013 siloxanes sludge 1 ngmL 1	Analyte	0.1	3.38	53111.6
11_2_2013 siloxanes sludge 5 ngmL 1	Analyte	0.1	3.38	58997.3
11_2_2013 siloxanes sludge 10 ngmL 1	Analyte	0.1	3.38	56486.1
11_2_2013 siloxanes sludge 50 ngmL 1	Analyte	0.1	3.38	54819.5
11_2_2013 siloxanes sludge blank 2	Blank	0.1	3.38	53085.3
11_2_2013 siloxanes sludge 01ngmL 2	Analyte	0.1	3.38	55119.9
11_2_2013 siloxanes sludge 05ngmL 2	Analyte	0.1	3.38	44852.7
11_2_2013 siloxanes sludge 1 ngmL 2	Analyte	0.1	3.38	58381.7
11_2_2013 siloxanes sludge 5 ngmL 2	Analyte	0.1	3.38	58827.3
11_2_2013 siloxanes sludge 10 ngmL 2	Analyte	0.1	3.38	58210.8
11_2_2013 siloxanes sludge 50 ngmL 2	Analyte	0.1	3.38	58967.6
11_2_2013 siloxanes sludge blank 3	Blank	0.1	3.38	50971.0
11_2_2013 siloxanes sludge 01ngmL 3	Analyte	0.1	3.38	50479.9
11_2_2013 siloxanes sludge 05ngmL 3	Analyte	0.1	3.38	53633.7
11_2_2013 siloxanes sludge 1 ngmL 3	Analyte	0.1	3.38	58162.8
11_2_2013 siloxanes sludge 5 ngmL 3	Analyte	0.1	3.38	58804.2
11_2_2013 siloxanes sludge 10 ngmL 3	Analyte	0.1	3.38	58985.6
11_2_2013 siloxanes sludge 50 ngmL 3	Analyte	0.1	3.38	57637.3

Table C-18: Data of M4Q QAQC analysis with split ratio 1:10 on 11/3/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
11_3_2013 siloxanes water blank1	Blank	0.1	3.39	14622.5
11_3_2013 siloxanes water 01ngmL 1	Standard	0.1	3.39	17348.9
11_3_2013 siloxanes water 05ngmL 1	Standard	0.1	3.39	17075.9
11_3_2013 siloxanes water 1 ngmL 1	Standard	0.1	3.39	17164.4
11_3_2013 siloxanes water 5 ngmL 1	Standard	0.1	3.39	18365.9
11_3_2013 siloxanes water 10 ngmL 1	Standard	0.1	3.39	19174.1
11_3_2013 siloxanes water 50 ngmL 1	Standard	0.1	3.4	19845.6
11_3_2013 siloxanes water blank 2	Blank	0.1	3.39	16561.5
11_3_2013 siloxanes water 01ngmL 2	Standard	0.1	3.39	16164.9
11_3_2013 siloxanes water 05ngmL 2	Standard	0.1	3.4	16050.7
11_3_2013 siloxanes water 1 ngmL 2	Standard	0.1	3.39	15706.1
11_3_2013 siloxanes water 5 ngmL 2	Standard	0.1	3.39	18832.9
11_3_2013 siloxanes water 10 ngmL 2	Standard	0.1	3.39	18307.4
11_3_2013 siloxanes water 50 ngmL 2	Standard	0.1	3.39	17747.7
11_3_2013 siloxanes water blank 3	Blank	0.1	3.39	16496.7
11_3_2013 siloxanes water 01ngmL 3	Standard	0.1	3.39	15859.2
11_3_2013 siloxanes water 05ngmL 3	Standard	0.1	3.39	16099.4
11_3_2013 siloxanes water 1 ngmL 3	Standard	0.1	3.39	16003.1
11_3_2013 siloxanes water 5 ngmL 3	Standard	0.1	3.4	16000.1
11_3_2013 siloxanes water 10 ngmL 3	Standard	0.1	3.39	16407.7
11_3_2013 siloxanes water 50 ngmL 3	Standard	0.1	3.39	16839.2
11_3_2013 siloxanes sludge blank1	Blank	0.1	3.39	44006.7
11_3_2013 siloxanes sludge 01ngmL 1	Analyte	0.1	3.4	39173.6
11_3_2013 siloxanes sludge 05ngmL 1	Analyte	0.1	3.4	48819.6
11_3_2013 siloxanes sludge 1 ngmL 1	Analyte	0.1	3.39	45137.8
11_3_2013 siloxanes sludge 5 ngmL 1	Analyte	0.1	3.39	52231.8
11_3_2013 siloxanes sludge 10 ngmL 1	Analyte	0.1	3.4	50790.2
11_3_2013 siloxanes sludge 50 ngmL 1	Analyte	0.1	3.39	48786.5
11_3_2013 siloxanes sludge blank 2	Blank	0.1	3.4	45004.4
11_3_2013 siloxanes sludge 01ngmL 2	Analyte	0.1	3.4	47003.3
11_3_2013 siloxanes sludge 05ngmL 2	Analyte	0.1	3.39	41104.9
11_3_2013 siloxanes sludge 1 ngmL 2	Analyte	0.1	3.39	52588.9
11_3_2013 siloxanes sludge 5 ngmL 2	Analyte	0.1	3.39	53849.5
11_3_2013 siloxanes sludge 10 ngmL 2	Analyte	0.1	3.39	46521.4
11_3_2013 siloxanes sludge 50 ngmL 2	Analyte	0.1	3.39	53847.3
11_3_2013 siloxanes sludge blank 3	Blank	0.1	3.39	48754.9

11_3_2013 siloxanes sludge 01ngmL 3	Analyte	0.1	3.39	49815.2
11_3_2013 siloxanes sludge 05ngmL 3	Analyte	0.1	3.39	32569.0
11_3_2013 siloxanes sludge 1 ngmL 3	Analyte	0.1	3.39	42874.6
11_3_2013 siloxanes sludge 5 ngmL 3	Analyte	0.1	3.39	48924.0
11_3_2013 siloxanes sludge 10 ngmL 3	Analyte	0.1	3.39	47735.5
11_3_2013 siloxanes sludge 50 ngmL 3	Analyte	0.1	3.39	46930.6

Table C-19: Data of M4Q QAQC analysis with split ratio 1:10 on 11/4/13

Name	Type	Std. Conc ($ng \cdot mL^{-1}$)	RT	Area
11_4_2013 siloxanes water blank1	Blank	0.1	3.38	12031.4
11_4_2013 siloxanes water 01ngmL 1	Standard	0.1	3.38	24924.6
11_4_2013 siloxanes water 05ngmL 1	Standard	0.1	3.38	21925.3
11_4_2013 siloxanes water 1 ngmL 1	Standard	0.1	3.38	21042.1
11_4_2013 siloxanes water 5 ngmL 1	Standard	0.1	3.38	23547.5
11_4_2013 siloxanes water 10 ngmL 1	Standard	0.1	3.38	21893.4
11_4_2013 siloxanes water 50 ngmL 1	Standard	0.1	3.38	21617.3
11_4_2013 siloxanes water blank 2	Blank	0.1	3.38	17703.4
11_4_2013 siloxanes water 01ngmL 2	Standard	0.1	3.38	24664.5
11_4_2013 siloxanes water 05ngmL 2	Standard	0.1	3.38	22902.3
11_4_2013 siloxanes water 1 ngmL 2	Standard	0.1	3.38	22156.6
11_4_2013 siloxanes water 5 ngmL 2	Standard	0.1	3.38	21278.8
11_4_2013 siloxanes water 10 ngmL 2	Standard	0.1	3.38	18601.0
11_4_2013 siloxanes water 50 ngmL 2	Standard	0.1	3.38	21277.7
11_4_2013 siloxanes water blank 3	Blank	0.1	3.38	19437.4
11_4_2013 siloxanes water 01ngmL 3	Standard	0.1	3.38	21095.0
11_4_2013 siloxanes water 05ngmL 3	Standard	0.1	3.38	22107.7
11_4_2013 siloxanes water 1 ngmL 3	Standard	0.1	3.38	22120.3
11_4_2013 siloxanes water 5 ngmL 3	Standard	0.1	3.38	22904.0
11_4_2013 siloxanes water 10 ngmL 3	Standard	0.1	3.38	20483.2
11_4_2013 siloxanes water 50 ngmL 3	Standard	0.1	3.38	21553.5
11_4_2013 siloxanes sludge blank1	Blank	0.1	3.38	22621.7
11_4_2013 siloxanes sludge 01ngmL 1	Analyte	0.1	3.38	32617.0
11_4_2013 siloxanes sludge 05ngmL 1	Analyte	0.1	3.38	35392.2
11_4_2013 siloxanes sludge 1 ngmL 1	Analyte	0.1	3.38	36589.6
11_4_2013 siloxanes sludge 5 ngmL 1	Analyte	0.1	3.38	36576.0
11_4_2013 siloxanes sludge 10 ngmL 1	Analyte	0.1	3.38	37258.5
11_4_2013 siloxanes sludge 50 ngmL 1	Analyte	0.1	3.38	36611.9
11_4_2013 siloxanes sludge blank 2	Blank	0.1	3.38	37358.1

11_4_2013 siloxanes sludge 01ngmL 2	Analyte	0.1	3.38	38898.6
11_4_2013 siloxanes sludge 05ngmL 2	Analyte	0.1	3.38	38676.1
11_4_2013 siloxanes sludge 1 ngmL 2	Analyte	0.1	3.38	38453.0
11_4_2013 siloxanes sludge 5 ngmL 2	Analyte	0.1	3.39	39039.4
11_4_2013 siloxanes sludge 10 ngmL 2	Analyte	0.1	3.38	39210.2
11_4_2013 siloxanes sludge 50 ngmL 2	Analyte	0.1	3.38	38443.3
11_4_2013 siloxanes sludge blank 3	Blank	0.1	3.38	39250.8
11_4_2013 siloxanes sludge 01ngmL 3	Analyte	0.1	3.39	39085.5
11_4_2013 siloxanes sludge 05ngmL 3	Analyte	0.1	3.38	36939.0
11_4_2013 siloxanes sludge 1 ngmL 3	Analyte	0.1	3.38	37297.1
11_4_2013 siloxanes sludge 5 ngmL 3	Analyte	0.1	3.39	36787.5
11_4_2013 siloxanes sludge 10 ngmL 3	Analyte	0.1	3.38	39452.6
11_4_2013 siloxanes sludge 50 ngmL 3	Analyte	0.1	3.38	47844.6

Table C-20: Data of M4Q Fort Collins and Loveland analysis with split ratio 1:10 on 11/16/13

Name	Type	Std. Conc	RT	Area
2013_11_16 Siloxanes water Blank 1	Water Blank	0.1	3.38	30108.0
2013_11_16 Siloxanes water 0_1ngmL	Water Standard	0.1	3.38	31551.9
2013_11_16 Siloxanes water 5 ngmL	Water Standard	0.1	3.39	32580.1
2013_11_16 Siloxanes water 50 ngmL	Water Standard	0.1	3.38	30755.9
2013_11_16 Siloxanes sludge Blank 2	Sludge Blank	0.1	3.39	40061.0
2013_11_16 Siloxanes sludge 0_1 ngmL	Sludge Standard	0.1	3.39	37634.6
2013_11_16 Siloxanes sludge 0_5 ngmL	Sludge Standard	0.1	3.39	43155.6
2013_11_16 Siloxanes sludge 1 ngmL	Sludge Standard	0.1	3.39	47320.4
2013_11_16 Siloxanes sludge 5 ngmL	Sludge Standard	0.1	3.39	34903.6
2013_11_16 Siloxanes sludge 10 ngmL	Sludge Standard	0.1	3.38	32152.3
2013_11_16 Siloxanes sludge 50 ngmL	Sludge Standard	0.1	3.38	34215.4
2013_11_16 Siloxanes water Blank 3	Blank	0.1	3.53	1070.1
2013_11_16 Siloxanes FC 11 Effluent	Water Analyte	0.1	3.38	299676.6
2013_11_16 Siloxanes FC 21 Influent	Water Analyte	0.1	3.38	74726.6
2013_11_16 Siloxanes FC 31 1sludge	Sludge Analyte	0.1	3.39	29201.5
2013_11_16 Siloxanes FC 41 WAS	Sludge Analyte	0.1	3.39	60771.8
2013_11_16 Siloxanes FC 51 Digested sludge	Sludge Analyte	0.1	3.38	39549.5
2013_11_16 Siloxanes water Blank 4	Blank	0.1	3.53	753.0
2013_11_16 Siloxanes FC 12 Effluent	Water Analyte	0.1	3.39	267886.3
2013_11_16 Siloxanes FC 22 Influent	Water Analyte	0.1	3.38	88254.7
2013_11_16 Siloxanes FC 32 1sludge	Sludge Analyte	0.1	3.39	30906.2
2013_11_16 Siloxanes FC 42 WAS	Sludge Analyte	0.1	3.39	54485.5
2013_11_16 Siloxanes FC 52 Digested	Sludge Analyte	0.1	3.39	32777.1

sludge				
2013_11_16 Siloxanes water Blank 5	Blank	0.1	3.55	683.0
2013_11_16 Siloxanes LL 11 Effluent	Water Analyte	0.1	3.38	287456.4
2013_11_16 Siloxanes LL 21 PEF	Water Analyte	0.1	3.38	117975.4
2013_11_16 Siloxanes LL 31 Primary Effluent	Water Analyte	0.1	3.38	80193.4
2013_11_16 Siloxanes LL 41 Influent	Water Analyte	0.1	3.38	77508.1
2013_11_16 Siloxanes LL 51 2 digester	Sludge Analyte	0.1	3.38	30989.7
2013_11_16 Siloxanes LL 61 1 digester	Sludge Analyte	0.1	3.39	24769.1
2013_11_16 Siloxanes LL 71 Primary sludge	Sludge Analyte	0.1	3.38	38035.1
2013_11_16 Siloxanes LL 81 WAS	Sludge Analyte	0.1	3.38	198843.5
2013_11_16 Siloxanes water Blank 6	Blank	0.1	3.39	240.2
2013_11_16 Siloxanes LL 12 Effluent	Water Analyte	0.1	3.38	258358.9
2013_11_16 Siloxanes LL 22 PEF	Water Analyte	0.1	3.38	102713.4
2013_11_16 Siloxanes LL 32 Primary Effluent	Water Analyte	0.1	3.38	79113.0
2013_11_16 Siloxanes LL 42 Influent	Water Analyte	0.1	3.38	65771.7
2013_11_16 Siloxanes LL 52 2 digester	Sludge Analyte	0.1	3.38	39257.6
2013_11_16 Siloxanes LL 62 1 digester	Sludge Analyte	0.1	3.38	24868.7
2013_11_16 Siloxanes LL 72 Primary sludge	Sludge Analyte	0.1	3.38	31912.0
2013_11_16 Siloxanes LL 82 WAS	Sludge Analyte	0.1	3.38	198092.4

Table C-21: Data of M4Q Boulder analysis with split ratio 1:10 on 11/22/13

Name	Type	Std. Conc	RT	Area
2013_11_22 Siloxanes water Blank 1	Blank	0.1	3.34	20354.0
2013_11_22 Siloxanes water 0_1ngmL	Standard	0.1	3.34	19370.2
2013_11_22 Siloxanes water 5 ngmL	Standard	0.1	3.33	17662.1
2013_11_22 Siloxanes water 50 ngmL	Standard	0.1	3.34	12646.3
2013_11_22 Siloxanes sludge Blank 2	Blank	0.1	3.34	36981.3
2013_11_22 Siloxanes sludge 0_1 ngmL	Standard	0.1	3.34	28608.6
2013_11_22Siloxanes sludge 0_5 ngmL	Standard	0.1	3.34	38472.4
2013_11_22 Siloxanes sludge 1 ngmL	Standard	0.1	3.34	22901.9
2013_11_22 Siloxanes sludge 5 ngmL	Standard	0.1	3.34	23311.9
2013_11_22 Siloxanes sludge 10 ngmL	Standard	0.1	3.34	30009.7
2013_11_22 Siloxanes sludge 50 ngmL	Standard	0.1	3.34	37965.4
2013_11_22 Siloxanes water Blank 3	Blank	0.1	3.35	218.0
2013_11_22 Siloxanes water Blank 4	Blank	0.1	3.51	618.5
2013_11_22 Siloxanes water Blank 5	Blank	0.1	3.36	198.3

2013_11_22 Siloxanes Rec 0_25ngmL1	Analyte	0.1	3.34	39520.2
2013_11_22 Siloxanes Rec 0_25ngmL2	Analyte	0.1	3.34	31740.4
2013_11_22 Siloxanes Rec 0_25ngmL3	Analyte	0.1	3.34	34014.9
2013_11_22 Siloxanes Rec 7_5 ngmL1	Analyte	0.1	3.34	38226.2
2013_11_22 Siloxanes Rec 7_5 ngmL2	Analyte	0.1	3.34	27769.7
2013_11_22 Siloxanes Rec 7_5 ngmL3	Analyte	0.1	3.34	38902.8
2013_11_22 Siloxanes Rec 25 ngmL 1	Analyte	0.1	3.34	34668.3
2013_11_22 Siloxanes Rec 25 ngmL 2	Analyte	0.1	3.34	35697.9
2013_11_22 Siloxanes Rec 25 ngmL 3	Analyte	0.1	3.34	33698.2
2013_11_22 Siloxanes BD Effluent 1	Analyte	0.1	3.34	243919.7
2013_11_22 Siloxanes BD Effluent 2	Analyte	0.1	3.34	181684.8
2013_11_22 Siloxanes BD Centrifuge 1	Analyte	0.1	3.34	45754.7
2013_11_22 Siloxanes BD Centrifuge 2	Analyte	0.1	3.34	48727.0
2013_11_22 Siloxanes BD Influent 1	Analyte	0.1	3.34	158708.7
2013_11_22 Siloxanes BD Influent 2	Analyte	0.1	3.34	146230.9
2013_11_22 Siloxanes BD Digester 1	Analyte	0.1	3.34	27552.8
2013_11_22 Siloxanes BD Digester 2	Analyte	0.1	3.34	24024.0
2013_11_22 Siloxanes BD WAS 1	Analyte	0.1	3.34	17896.1
2013_11_22 Siloxanes BD WAS 2	Analyte	0.1	3.34	14213.0
2013_11_22 Siloxanes Primary sludge 1	Analyte	0.1	3.34	18287.2
2013_11_22 Siloxanes Primary sludge 2	Analyte	0.1	3.34	14993.2

Table C-22: Data of M4Q Fort Collins and Loveland analysis with split ratio 1:10 on 1/10/14

Name	Type	Std. Conc	RT	Area
2014_1_10 siloxane blank1 water	Blank	0.25		
2014_1_10 siloxane 05D405D5water	Standard	0.25	3.37	49026.3
2014_1_10 siloxane 1D45D5 water	Standard	0.25	3.37	41967.0
2014_1_10 siloxane 5D450D5water	Standard	0.25	3.37	31788.4
2014_1_10 siloxane 15D4150D5 water	Standard	0.25	3.37	34942.2
2014_1_10 siloxane blank2 sludge	Blank	0.25	3.37	15894.2
2014_1_10 siloxane 05D405D5sludge	Standard	0.25	3.37	13303.9
2014_1_10 siloxane 1D45D5 sludge	Standard	0.25	3.37	13714.9
2014_1_10 siloxane 5D450D5sludge	Standard	0.25	3.37	14887.7
2014_1_10 siloxane 15D4150D5 sludge	Standard	0.25	3.37	15391.8
2014_1_10 siloxane blank3 water	Blank	0.25	3.71	302.5
2014_1_10 siloxane blank4 water	Blank	0.25	3.37	138.1
2014_1_10 siloxane blank5 water	Blank	0.25	3.46	344.9
2014_1_10 siloxane blank6 water	Blank	0.25	3.38	150.7
2014_1_10 siloxane FC effluent1	Analyte	0.25	3.37	243043.8

2014_1_10 siloxane FC effluent2	Analyte	0.25	3.37	189360.2
2014_1_10 siloxane FCinfluent1	Analyte	0.25	3.37	62529.6
2014_1_10 siloxane FCinfluent2	Analyte	0.25	3.37	63879.6
2014_1_10 siloxane FCcentrate1	Analyte	0.25	3.37	137953.4
2014_1_10 siloxane FCcentrate2	Analyte	0.25	3.37	157637.0
2014_1_10 siloxane FC primary sludge1	Analyte	0.25	3.37	34009.8
2014_1_10 siloxane FC primary sludge2	Analyte	0.25	3.37	23386.8
2014_1_10 siloxane FC WAS1	Analyte	0.25	3.37	27078.4
2014_1_10 siloxane FC WAS2	Analyte	0.25	3.37	13313.7
2014_1_10 siloxane FC RAS 1	Analyte	0.25	3.37	31717.4
2014_1_10 siloxane FC RAS 2	Analyte	0.25	3.37	42680.3
2014_1_10 siloxane FC anaerobic sludge1	Analyte	0.25	3.37	19239.3
2014_1_10 siloxane FC anaerobic sludge2	Analyte	0.25	3.37	13969.3
2014_1_10 siloxane LL effluent 1	Analyte	0.25	3.37	163411.6
2014_1_10 siloxane LL effluent 2	Analyte	0.25	3.37	228104.6
2014_1_10 siloxane LL primary effluent 1	Analyte	0.25	3.37	66849.8
2014_1_10 siloxane LL primary effluent 2	Analyte	0.25	3.37	73852.6
2014_1_10 siloxane LL influent 1	Analyte	0.25	3.37	93275.8
2014_1_10 siloxane LL influent 2	Analyte	0.25	3.37	92447.5
2014_1_10 siloxane LL Centrate 1	Analyte	0.25	3.37	63037.8
2014_1_10 siloxane LL Centrate 2	Analyte	0.25	3.37	71914.7
2014_1_10 siloxane LL primary digester 1	Analyte	0.25	3.37	14608.0
2014_1_10 siloxane LL primary digester 2	Analyte	0.25	3.37	14142.6
2014_1_10 siloxane LL RAS 1	Analyte	0.25	3.37	115406.3
2014_1_10 siloxane LL RAS 2	Analyte	0.25	3.37	102829.0
2014_1_10 siloxane LL Primary sludge 1	Analyte	0.25	3.37	18655.9
2014_1_10 siloxane LL Primary sludge 2	Analyte	0.25	3.37	19189.3

Table C-23: Data of M4Q Fort Collins and Loveland analysis with split ratio 1:10 on 2/5/14

Name	Type	Std. Conc	RT	Area
2014_2_5 siloxane blank1 water	Blank	0.25	3.36	17110.8
2014_2_5 siloxane 05D405D5water	Standard	0.25	3.35	17507.1
2014_2_5 siloxane 1D45D5 water	Standard	0.25	3.35	17755.6
2014_2_5 siloxane 5D450D5water	Standard	0.25	3.35	16875.0
2014_2_5 siloxane 15D4150D5 water	Standard	0.25	3.35	17958.9
2014_2_5 siloxane blank2 sludge	Blank	0.25	3.34	19128.3
2014_2_5 siloxane 05D405D5sludge	Standard	0.25	3.34	18541.0
2014_2_5 siloxane 1D45D5 sludge	Standard	0.25	3.35	18615.9

2014_2_5 siloxane 5D450D5sludge	Standard	0.25	3.35	18217.1
2014_2_5 siloxane 15D4150D5 sludge	Standard	0.25	3.49	18424.5
2014_2_5 siloxane blank3 water	Blank	0.25		
2014_2_5 siloxane blank4 water	Blank	0.25		
2014_2_6 siloxanes Blanck water 5	Analyte	0.25	3.36	29397.5
2014_2_5 siloxane Blank sludge	Blank	0.25	3.49	7677.9
2014_2_5 siloxane LL effluent 1	Analyte	0.25	3.35	146215.0
2014_2_5 siloxane LL effluent 2	Analyte	0.25	3.35	145964.5
2014_2_5 siloxane LL primary effluent 1	Analyte	0.25	3.35	31682.3
2014_2_5 siloxane LL primary effluent 2	Analyte	0.25	3.35	34490.9
2014_2_5 siloxane LL influent 1	Analyte	0.25	3.35	31331.3
2014_2_5 siloxane LL influent 2	Analyte	0.25	3.35	33950.4
2014_2_5 siloxane LL Centrate 1	Analyte	0.25	3.35	66076.9
2014_2_5 siloxane LL Centrate 2	Analyte	0.25	3.35	66266.5
2014_2_5 siloxane LL primary digester 1	Analyte	0.25	3.35	20832.9
2014_2_5 siloxane LL primary digester 2	Analyte	0.25	3.35	20886.7
2014_2_5 siloxane LL primary sludge 1	Analyte	0.25	3.36	6766.1
2014_2_5 siloxane LL Primary sludge 2	Analyte	0.25	3.35	6791.2
2014_2_5 siloxane LL secondary sludge 1	Analyte	0.25	3.35	119396.7
2014_2_5 siloxane LL secondary sludge2	Analyte	0.25	3.34	128624.6
2014_2_5 siloxane LL Primary clarifier 1	Analyte	0.25	3.35	23240.8
2014_2_5 siloxane LL Primary clarifier 2	Analyte	0.25	3.34	24333.8
2014_2_6 siloxanes FC eff 1	Analyte	0.25	3.36	118488.9
2014_2_6 siloxanes FC eff 2	Analyte	0.25	3.36	118520.4
2014_2_6 siloxanes FC inf 1	Analyte	0.25	3.36	26488.0
2014_2_6 siloxanes FC inf 2	Analyte	0.25	3.36	30487.6
2014_2_6 siloxanes FC Cen 1	Analyte	0.25	3.36	55484.9
2014_2_6 siloxanes FC Cen 2	Analyte	0.25	3.36	51443.8
2014_2_6 siloxanes FC Pri sludge 1	Analyte	0.25	3.36	10088.1
2014_2_6 siloxanes FC Pri sludge 2	Analyte	0.25	3.37	11170.0
2014_2_6 siloxanes FC TWAS 1	Analyte	0.25	3.36	20235.2
2014_2_6 siloxanes FC TWAS 2	Analyte	0.25	3.36	19984.9
2014_2_6 siloxanes FC RAS 1	Analyte	0.25	3.36	37941.5
2014_2_6 siloxanes FC RAS 2	Analyte	0.25	3.36	37995.8
2014_2_6 siloxanes FC Anaerobic Sludge 1	Analyte	0.25	3.51	27165.9
2014_2_6 siloxanes FC Anaerobic Sludge 2	Analyte	0.25	3.37	27614.8

Table C-24: Data of M4Q Fort Collins and Loveland analysis with split ratio 1:10 on 3/5/14

Name	Type	Std. Conc	RT	Area
2014_3_5 siloxanes Blank 1	Blank	0.25	3.35	16699.7
2014_3_5 siloxanes water 05D405D5	Standard	0.25	3.34	16949.9
2014_3_5 siloxanes water 1D45D5	Standard	0.25	3.34	15297.2
2014_3_5 siloxanes water 5D450D5	Standard	0.25	3.34	13120.4
2014_3_5 siloxanes water15D4150D5	Standard	0.25	3.34	14407.1
2014_3_5 siloxanes sludge Blank 2	Blank	0.25	3.35	18175.3
2014_3_5 siloxanes sludge 05D405D5	Standard	0.25	3.35	20127.0
2014_3_5 siloxanes sludge 1D45D5	Standard	0.25	3.35	26697.9
2014_3_5 siloxanes sludge 5D450D5	Standard	0.25	3.35	22715.5
2014_3_5 siloxanes sludge15D4150D5	Standard	0.25	3.35	21410.0
2014_3_5 siloxanes water Blank 3	Blank	0.25	3.51	1515.3
2014_3_5 siloxanes water Blank 4	Blank	0.25	3.51	1359.7
2014_3_6 siloxanes FC water blank1	Blank	0.25	3.51	1405.2
2014_3_6 siloxanes FC water blank 2	Blank	0.25	3.5	1209.7
2014_3_5 siloxanes LL effluent1	Analyte	0.25	3.35	122465.5
2014_3_5 siloxanes LL effluent 2	Analyte	0.25	3.35	118026.8
2014_3_5 siloxanes LL PEF 1	Analyte	0.25	3.35	24334.1
2014_3_5 siloxanes LL PEF 2	Analyte	0.25	3.35	21505.4
2014_3_5 siloxanes LL Influent 1	Analyte	0.25	3.35	23094.0
2014_3_5 siloxanes LL Influent 2	Analyte	0.25	3.34	20459.4
2014_3_5 siloxanes LL RAS 1	Analyte	0.25	3.35	51824.1
2014_3_5 siloxanes LL RAS 2	Analyte	0.25	3.35	48458.7
2014_3_5 siloxanes LL Primary Digester 1	Analyte	0.25	3.35	18112.6
2014_3_5 siloxanes LL Primary Digester 2	Analyte	0.25	3.35	17426.8
2014_3_5 siloxanes LL Primary sludge 1	Analyte	0.25	3.35	6093.4
2014_3_5 siloxanes LL Primary sludge 2	Analyte	0.25	3.35	6498.2
2014_3_6 siloxanes FC effluent 1	Analyte	0.25	3.36	15472.6
2014_3_6 siloxanes FC effluent 2	Analyte	0.25	3.36	15742.2
2014_3_6 siloxanes FC influent 1	Analyte	0.25	3.36	8581.6
2014_3_6 siloxanes FC influent 2	Analyte	0.25	3.36	8543.3
2014_3_6 siloxanes FC centrate 1	Analyte	0.25	3.36	44062.0
2014_3_6 siloxanes FC centrate 2	Analyte	0.25	3.36	34363.4
2014_3_6 siloxanes FC Primary sludge 1	Analyte	0.25	3.36	7747.5
2014_3_6 siloxanes FC Primary sludge 2	Analyte	0.25	3.36	5931.2
2014_3_6 siloxanes FC TWAS 1	Analyte	0.25	3.36	10658.0
2014_3_6 siloxanes FC TWAS 2	Analyte	0.25	3.36	11222.2
2014_3_6 siloxanes FC RAS 1	Analyte	0.25	3.36	23545.7

2014_3_6 siloxanes FC RAS 2	Analyte	0.25	3.36	24603.7
2014_3_6 siloxanes FC Anaerobic sludge 1	Analyte	0.25	3.36	13760.4
2014_3_6 siloxanes FC Anaerobic sludge 2	Analyte	0.25	3.36	11516.0

Table C-25: Data of M4Q Fort Collins and Loveland analysis with split ratio 1:10 on 4/16/14

Name	Type	Std. Conc	RT	Area
2014_4_16 siloxanes water 05D405D5	Standard	0.25	3.34	5408.077
2014_4_16 siloxanes water 1D45D5	Standard	0.25	3.34	5327.765
2014_4_16 siloxanes water 5D450D5	Standard	0.25	3.34	5452.361
2014_4_16 siloxanes water 15D4150D5	Standard	0.25	3.34	5066.395
2014_4_16 siloxanes sludge 05D405D5	Standard	0.25	3.34	7515.253
2014_4_16 siloxanes sludge 1D45D5	Standard	0.25	3.34	8296.702
2014_4_16 siloxanes sludge 5D450D5	Standard	0.25	3.34	7414.271
2014_4_16 siloxanes sludge 15D4150D5	Standard	0.25	3.34	8580.272
2014_4_16 siloxanes blank water 1	Blank	0.25	3.34	7845.779
2014_4_16 silxoanes blank sludge 2	Blank	0.25	3.34	7268.543
2014_4_16 siloxanes Water blank 3	Blank	0.25	3.49	621.392
2014_4_16 siloxanes Water blank 4	Blank	0.25	3.46	931.769
2014_4_16 siloxanes water blank 5	Blank	0.25	3.47	805.72
2014_4_16 siloxanes water blank 6	Blank	0.25	3.47	675.165
2014_4_16 siloxanes FC effluent 1	Analyte	0.25	3.33	56907.72
2014_4_16 siloxanes FC effluent 2	Analyte	0.25	3.33	62584.11
2014_4_16 siloxanes FC Influent 1	Analyte	0.25	3.33	9774.847
2014_4_16 siloxanes FC Influent 2	Analyte	0.25	3.33	9247.812
2014_4_16 siloxanes FC Centrate 1	Analyte	0.25	3.34	27878.51
2014_4_16 siloxanes FC Centrate 2	Analyte	0.25	3.33	23104.38
2014_4_16 siloxanes FC Primary sludge 1	Analyte	0.25	3.34	4419.066
2014_4_16 siloxanes FC Primary sludge 2	Analyte	0.25	3.34	5316.127
2014_4_16 siloxanes FC TWAS 1	Analyte	0.25	3.34	8751.543
2014_4_16 siloxanes FC TWAS 2	Analyte	0.25	3.44	507.474
2014_4_16 siloxanes FC RAS 1	Analyte	0.25	3.34	14792.4
2014_4_16 siloxanes FC RAS 2	Analyte	0.25	3.34	16085.58
2014_4_16 siloxanes FC Anaerobic sludge 1	Analyte	0.25	3.33	8139.403
2014_4_16 siloxanes FC Anaerobic sludge 2	Analyte	0.25	3.34	9167.263
2014_4_16 siloxanes LL effluent 1	Analyte	0.25	3.33	52542.35
2014_4_16 siloxanes LL effluent 2	Analyte	0.25	3.33	40801.04
2014_4_16 siloxanes LL primary effluent 1	Analyte	0.25	3.33	14656.07
2014_4_16 siloxanes LL primary effluent 2	Analyte	0.25	3.33	14082.34
2014_4_16 siloxanes LL influent 1	Analyte	0.25	3.33	8456.379
2014_4_16 siloxanes LL influent 2	Analyte	0.25	3.33	6691.639

2014_4_16 siloxanes LL Centrate 1	Analyte	0.25	3.33	21911.28
2014_4_16 siloxanes LL Centrate 2	Analyte	0.25	3.33	22687.53
2014_4_16 siloxanes LL RAS1	Analyte	0.25	3.33	19306.93
2014_4_16 siloxanes LL RAS 2	Analyte	0.25	3.34	16584.22
2014_4_16 siloxanes LL Primary digestion 1	Analyte	0.25	3.34	7111.033
2014_4_16 siloxanes LL Primary digestion 2	Analyte	0.25	3.34	7616.066
2014_4_16 siloxanes LL primary sludge 1	Analyte	0.25	3.34	3669.783
2014_4_16 siloxanes LL primary sludge 2	Analyte	0.25	3.33	4376.417

Table C-26: Data of M4Q Fort Collins and Loveland analysis with split ratio 1:10 on 5/20/14

Name	Type	Std. Conc	RT	Area
2014_5_20 siloxane blank water 1	Blank	0.25	3.31	12778.15
2014_5_20 siloxane water 0505ngmL	Standard	0.25	3.31	14169.85
2014_5_20 siloxane water D41D55ngmL	Standard	0.25	3.31	15792.52
2014_5_20 siloxane water D45D550ngmL	Standard	0.25	3.31	12931.41
2014_5_20 siloxane water D415D5150ngmL	Standard	0.25	3.31	13711.44
2014_5_21 siloxanes sludge blank	Blank	0.25	3.31	24628.78
2014_5_20 siloxane sludge D405D505ngmL	Standard	0.25	3.31	45872.97
2014_5_20 siloxane sludge D41D55ngmL	Standard	0.25	3.31	37667.94
2014_5_20 siloxane sludge D45D550ngmL	Standard	0.25	3.31	59086.42
2014_5_20 siloxane sludge D415D5150ngmL	Standard	0.25	3.31	49440.32
2014_5_20 siloxane blank water 00	Blank	0.25	3.31	372.682
2014_5_20 siloxane blank water 2	Blank	0.25	3.25	687.804
2014_5_20 siloxane blank water 3	Blank	0.25	3.28	608.489
2014_5_20 siloxane blank water 4	Blank	0.25	3.3	371.491
2014_5_20 siloxane blank water 5	Blank	0.25	3.31	264.11
2014_5_20 siloxane FC Effluent 1	Analyte	0.25	3.31	202338.5
2014_5_20 siloxane FC Effluent 2	Analyte	0.25	3.31	259076
2014_5_20 siloxane FC influent 1	Analyte	0.25	3.31	73743.65
2014_5_20 siloxane FC influent 2	Analyte	0.25	3.31	63900.12
2014_5_20 siloxane FC centrate 1	Analyte	0.25	3.31	122786.8
2014_5_20 siloxane FC centrate 2	Analyte	0.25	3.31	108948.7
2014_5_20 siloxane FC primary sludge 1	Analyte	0.25	3.31	19658.73
2014_5_20 siloxane FC primary sludge 2	Analyte	0.25	3.31	26128.56
2014_5_20 siloxane FC TWAS 1	Analyte	0.25	3.31	24842.17
2014_5_20 siloxane FC TWAS 2	Analyte	0.25	3.31	20624.5
2014_5_20 siloxane FC RAS 1	Analyte	0.25	3.31	85945.52
2014_5_20 siloxane FC RAS 2	Analyte	0.25	3.31	89509.54
2014_5_20 siloxane FC Digester 1	Analyte	0.25	3.31	52401.52
2014_5_20 siloxane FC Digester 2	Analyte	0.25	3.31	51633.67

2014_5_20 siloxane LL Effluent 1	Analyte	0.25	3.31	153673.3
2014_5_20 siloxane LL Effluent 2	Analyte	0.25	3.31	106542.4
2014_5_20 siloxane LL Primary effluent 1	Analyte	0.25	3.31	63197.65
2014_5_20 siloxane LL Primary effluent 2	Analyte	0.25	3.31	62415.07
2014_5_20 siloxane LL influent 1	Analyte	0.25	3.31	69779.58
2014_5_20 siloxane LL influent 2	Analyte	0.25	3.31	51525.83
2014_5_20 siloxane LL Centrate 1	Analyte	0.25	3.31	71158.3
2014_5_20 siloxane LL Centrate 2	Analyte	0.25	3.31	64682.64
2014_5_20 siloxane LL RAS 1	Analyte	0.25	3.31	19986.33
2014_5_20 siloxane LL RAS 2	Analyte	0.25	3.31	17685.56
2014_5_20 siloxane LL Digester 1	Analyte	0.25	3.31	24529.68
2014_5_20 siloxane LL Digester 2	Analyte	0.25	3.31	24584.71
2014_5_20 siloxane LL Primary sludge 1	Analyte	0.25	3.31	14624.15
2014_5_20 siloxane LL Primary sludge 2	Analyte	0.25	3.31	12877.23

Table C-27: Data of M4Q Volume of Acetone Impact on 5/20/2014

Name	Type	Std. Conc	RT	Area
2014_5_20 siloxane acetone 50uL water	Analyte	0.25	3.31	15118.05
2014_5_20 siloxane acetone 100uL water	Analyte	0.25	3.31	22576.25
2014_5_20 siloxane acetone 500uL water	Analyte	0.25	3.27	425867.5
2014_5_20 siloxane acetone 1000uL water	Analyte	0.25	3.31	795927.2
2014_5_20 siloxane acetone 1500uL water	Analyte	0.25	3.26	545047.6
2014_5_20 siloxane acetone 1500uL water2	Analyte	0.25	3.27	465717.5
2014_5_20 siloxane acetone 50uL sludge	Analyte	0.25	3.31	50732.5
2014_5_20 siloxane acetone 100uL sludge	Analyte	0.25	3.31	47688.41
2014_5_20 siloxane acetone 500uL sludge	Analyte	0.25	3.3	69423.43
2014_5_20 siloxane acetone 1000uL sludge	Analyte	0.25	3.29	53668.13
2014_5_20 siloxane acetone 1500uL sludge	Analyte	0.25	3.28	90606.3
2014_5_20 siloxane acetone 1500uL sludge2	Analyte	0.25	3.28	73830.37

D. Total Solids Analysis

Table D-1: Data of TS for Fort Collins and Loveland analysis on 1/16/2014

		Trial 1				Trial 2				
Location	Name	Weight of dish (g)	Weight after dry (g)	Volume (mL)	TS (mg/L)	Weight of dish (g)	Weight after dry (g)	Volume (mL)	TS (mg/L)	Avg. TS (mg/L)
Loveland	RTD Sludge	1.3137	1.7623	10	44860	1.3076	1.873	10	56540	50700
	Primary Sludge	1.3183	1.4224	5	20820			0		20820
	RAS	1.3069	1.3467	10	3980	1.3161	1.3593	10	4320	4150
	Primary Digester	1.3101	1.391	5	16180			0		16180
Fort Collins	TWAS	1.3073	1.7691	10	46180	1.31	1.7664	10	45640	45910
	Primary Sludge	1.303	1.5135	5	42100	1.3022	1.875	10	57280	49690
	RAS	1.3129	1.4147	10	10180	1.309	1.4066	10	9760	9970
	Anaerobic Digester	1.3146	1.4934	10	17880	1.3102	1.4915	10	18130	18005

Table D-2: Data of TS for Fort Collins and Loveland analysis on 2/5/2014

		Trial 1		Trial 2			
		Dish weight 1 (g)	Total Dry weight 1 (g)	Dish weight 2 (g)	Total Dry weight 2 (g)	Sample volume (mL)	Average TS (mg/L)
Loveland	RTD	1.1678	1.216	1.1971	1.2427	5	9380
	Secondary Sludge	1.1717	1.1901	1.1804		5	1840
	Primary Digester	1.1756	1.2486	1.1783	1.2522	5	14690
	Primary Sludge	1.179	1.3813	1.1783	1.3724	5	39640
Fort Collins	Centrated	1.1744	1.1915	1.1645	1.1809	10	1675
	Digested	1.1682	1.3397	1.1801	1.36	10	17570
	RAS	1.1778	1.2449	1.178	1.2833	10	8620
	TWAS	1.1801	1.4938	1.1826	1.4788	10	30495
	Primary Sludge	1.3151	1.6399	1.3051	1.6468	10	33325

Table D-3: Data of TS for Fort Collins and Loveland analysis on 3/6/2014

		Trial 1		Trial 2			
		Plate weight 1 (g)	Total dry weight 1 (g)	Plate weight 2 (g)	Total dry weight 2 (g)	Sludge volume (mL)	Avg. TS (mg/L)
Loveland	RAS	1.1874	1.2362	1.1826	1.2314	10	4880
	Primary Digester	1.182	1.3343	1.1797	1.3277	10	15015
	Primary Sludge	1.1905	1.317	1.1667	1.4245	10	19215
Fort Collins	RAS	1.1778	1.2559	1.1859	1.2625	10	7735
	Anaerobic Digester	1.1894	1.4012	1.1801	1.3834	10	20755
	Primary Sludge	1.1781	1.553	1.1783	1.422	10	30930
	TWAS	1.1699	1.5105	1.1841	1.5762	10	36635

Table D-4: Data of TS for Fort Collins and Loveland analysis on 4/16/2014

		Trial 1		Trial 2			
		Plate weight 1 (g)	Total dry weight 1 (g)	Plate weight 2 (g)	Total dry weight 2 (g)	Sludge volume (mL)	Avg. TS (mg/L)
Loveland	RAS	1.1757	1.2263	1.1788	1.2302	10	5100
	Digester Sludge	1.1854	1.3339	1.1806	1.3264	10	14715
	Primary Sludge	1.181	1.382	1.1827	1.4317	10	22500
Fort Collins	RAS	1.312	1.389	1.3059	1.3806	10	7585
	Digester Sludge	1.3079	1.4922	1.3119	1.505	10	18870
	TWAS	1.1785	1.4853	1.179	1.5351	10	33145
	Primary Sludge	1.1922	1.5381	1.182	1.5247	10	34430

Table D-5: Data of TS for Fort Collins and Loveland analysis on 5/20/2014

		Trial 1		Trial 2			
		Dish weight 1	Total dry weight1	Dish weight 2	Total dry weight2	volume(mL)	TS(mg/L)
Fort Collins	TWAS	1.1741	1.5074	1.1847	1.5351	10	34185
	Primary	1.185	1.34	1.1823	1.3599	10	16630

	Sludge						
	RAS	1.1786	1.2173	1.1603	1.2023	5	8070
	Digester Sludge	1.1617	1.2425	1.183	1.2628	5	16060
Loveland	RAS	1.1807	1.4442	1.1711	1.4381	10	26525
	Primary Sludge	1.1784	1.556	1.17	1.4875	10	34755
	Digester Sludge	1.1623	1.3122	1.1805	1.33	10	14970

E. Awareness Survey

An online survey about the awareness of presence of siloxanes among employees in WWTP in the US was conducted by the Qualtrics.com. Utility managers, plant operators, engineers, plant support and project manager were involved in this questionnaire. The Figure E-1 indicates the survey questionare and logics. 24 responds were obtained. The following Table E-1 shows the survey report.

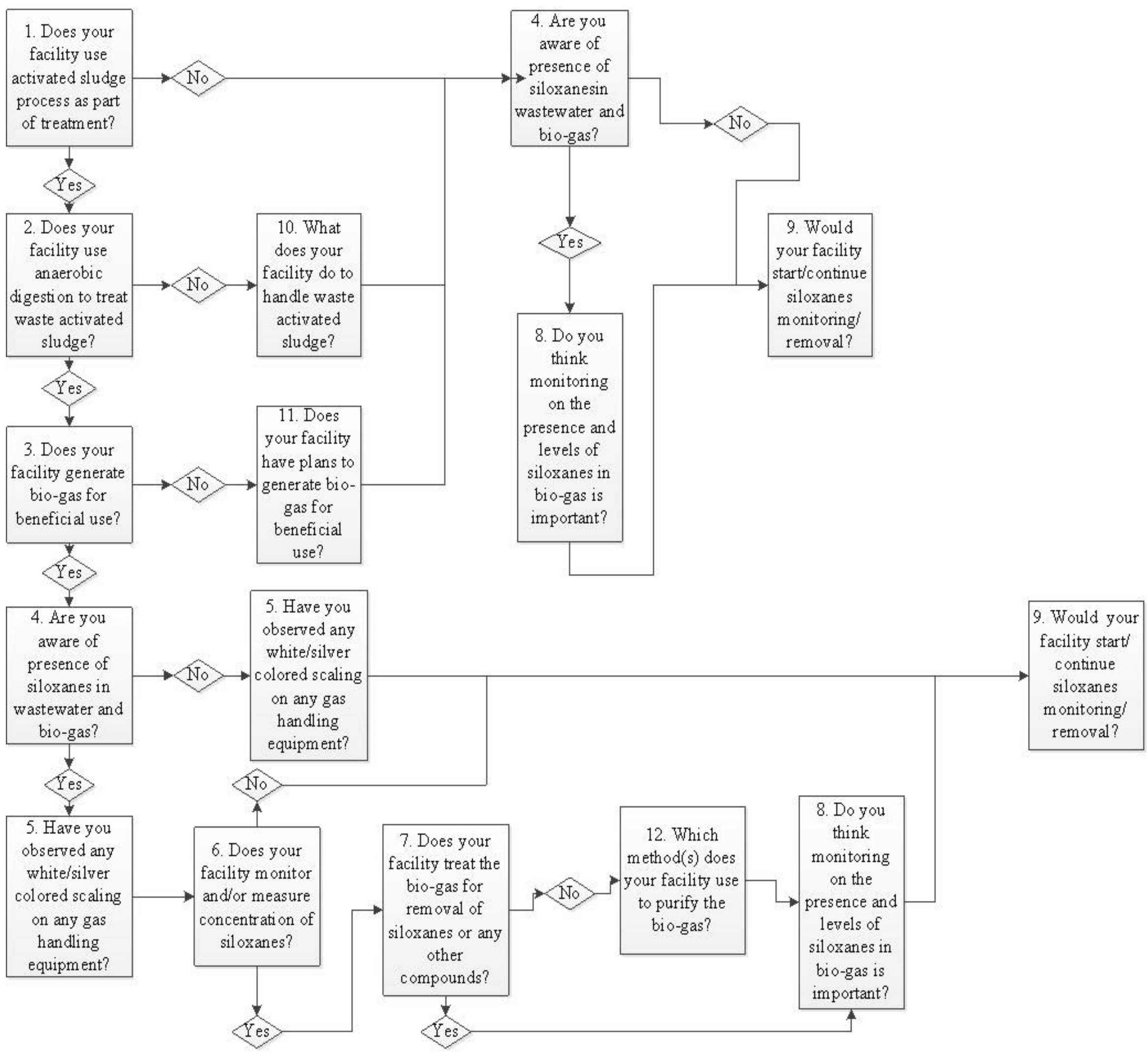


Figure E-1: Awareness Survey Questionnare and Logics

Table E-1: Awareness Survey Results

		Yes	No	Total
1	Does your facility use activated sludge process as part of treatment?	12	2	14
2	Does your facility use anaerobic digestion to treat waste activated sludge?	7	5	12
3	Does your facility generate bio-gas for beneficial use?	7	0	7
4	Are you aware of presence of siloxanes in wastewater and bio-gas?	10	4	14
5	Have you observed any white/silver colored scaling on any gas handling equipment?	6	2	8
6	Does your facility monitor and/or measure concentration of siloxanes?	1	6	7
7	Does your facility treat the bio-gas for removal of siloxanes or any other compounds?	1	0	1
8	Do you think monitoring on the presence and levels of siloxanes in bio-gas is important?	4	1	5
9	Would your facility start/continue siloxanes monitoring/removal?	5	9	14

The Figure E-2 shows the answers of the utilities generate biogas. About 86% utilities use activated sludge process but only 58% of these ones use anaerobic digestion to treat waste activated sludge and generate bio-gas for beneficial use. About 71% of them aware the occurrence of siloxanes in WWTPs but only 36% of them would start or continue silxoanes monitoring or removal.

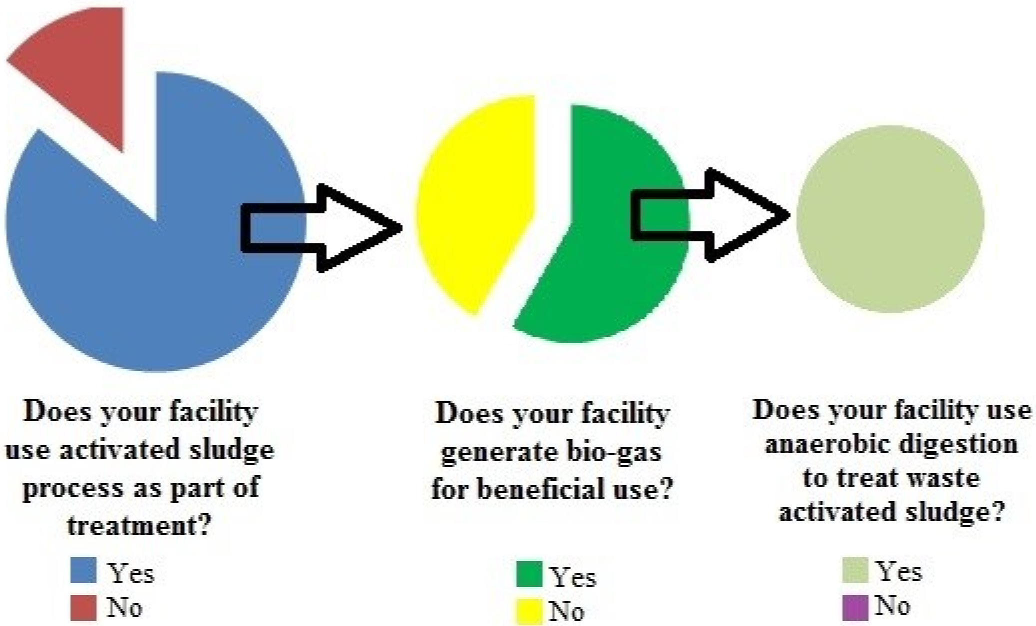


Figure E-2: Results of Generating Biogas.

F. Sludge Picture

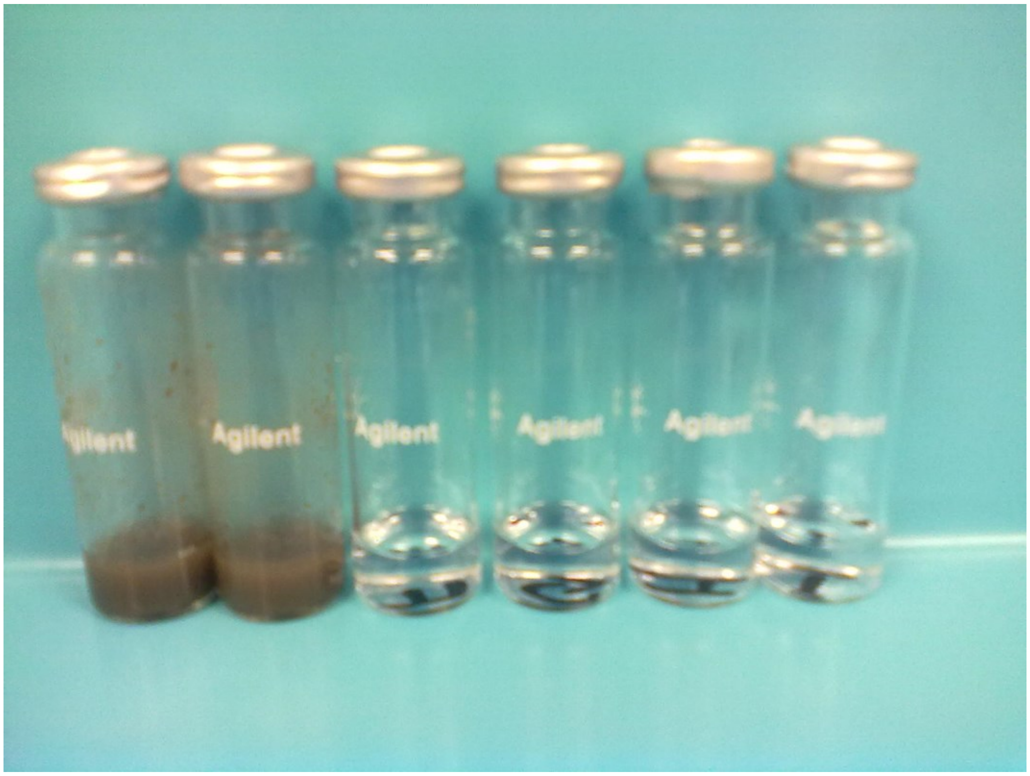


Figure F-1: Headspace Vials with Samples (Left 2: Sludge, Right 4: Wastewater)

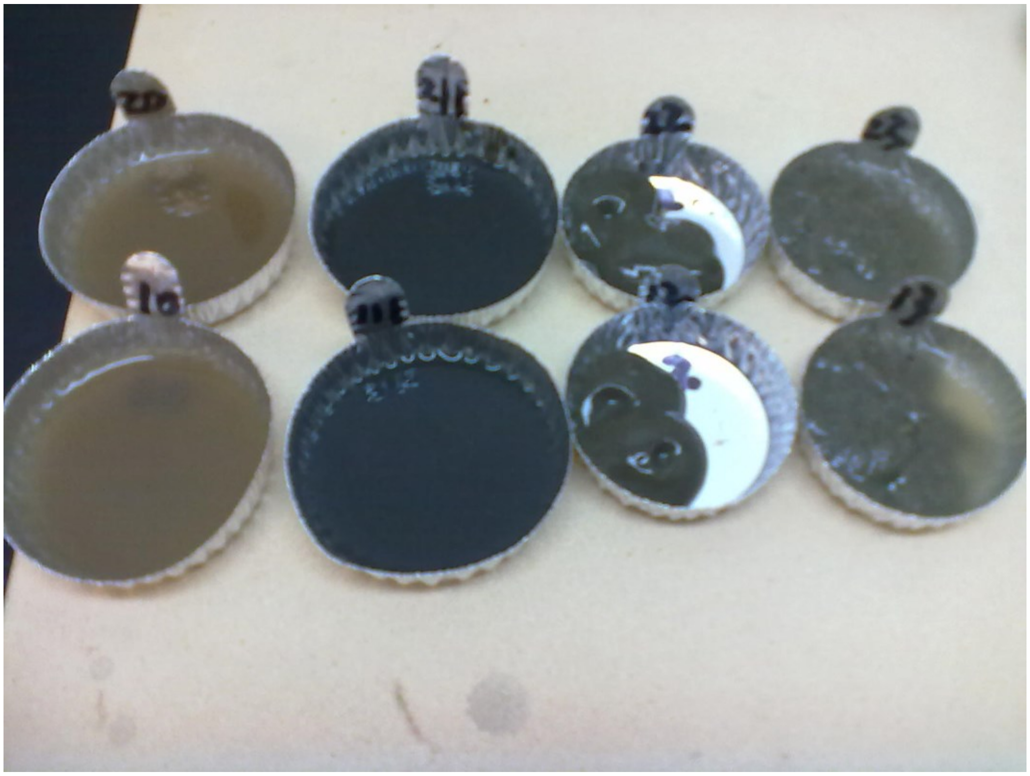


Figure F-2: Sludge Samples for TS Analysis (From Left to Right: RAS, Digester Sludge, TWAS, Primary Sludge)

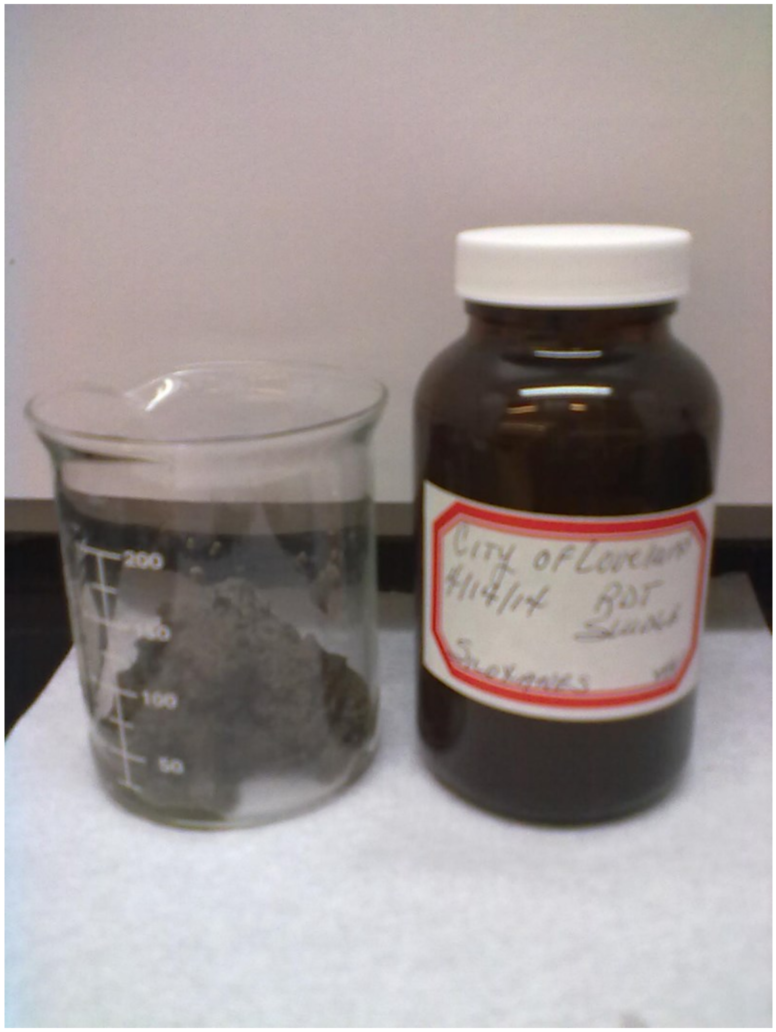


Figure F-3: RDT Sludge from City of Loveland

Figure F-4: Discharge Point of Drake WWRF

Figure F-5: Discharge point of Loveland WWTP