SILOXANES AND OTHER HARMFUL CONTAMINANTS: THEIR IMPORTANCE IN TOTAL LFG QUALITY MANAGEMENT

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Abstract

Total LFG (landfill gas) quality management (QM) is a relatively new concept that was introduced by this company. The primary premise of total LFGQM is that there is more than one dimension to properly treating landfill gas for use as a fuel and that these must all be addressed. First, there is the need to remove harmful organics, like the siloxanes. Next, in many applications, it is necessary to control the moisture content, then the sulfur content, and perhaps even halogenated species (like vinyl chloride), as well. LFG treatment can involve as many as 8 or more individual treatment processes, depending on the ultimate use of the gas.

Until recently, the equipment required for the individual treatment steps for gas conditioning and siloxane removal were supplied by different companies. This made it difficult for operators to manage gas quality and maintenance issues properly because there was little or no coordination between the processes. By performing a comprehensive gas analysis, and using the results, it is possible to engineer a complete gas treatment process that will not only perform all of the needed treatment steps, but also to obtain this from one source instead of many. Now, technical assistance for the entire process is available with one phone call.

This paper will explore what is involved in the gas testing, how this information is used to design a treatment system, and the critical parameters that have to be addressed for several gas applications.

Summary

Over the past several years, a lot of attention has been focused on siloxanes in landfill gas (LFG) and the costly problems they create for operators of power generation equipment. While it is important to focus on siloxanes, it is equally important to focus on other parameters of the gas to properly design a treatment process.

The need for knowing what is in LFG is necessary due to the widely varying types of waste landfills accept (or have accepted in the past). Multiple-use landfills that accept garbage, construction and industrial wastes will have much different LFG contaminant profiles than those of bedroom communities who collect garbage only. Moreover, although environmental laws now exist to prevent disposal of toxic or hazardous chemicals in landfills, this practice was common in the past. Illegal dumping of toxic chemicals into landfills is also possible. The VOC profile of a landfill results partly from the decomposition of organic matter and partly from the volatile nature of contaminants that reside in the landfill. For example, low molecular weight contaminants like ethanol and even high molecular weight contaminants like carene and d-limonene result from the decay of organic matter while contaminants like naphthalene result from chemicals residing in the landfill.

In order to develop an accurate profile of any LFG, comprehensive testing must be performed. A complete battery of the needed tests includes major gas components, total identification and speciation of the VOCs (volatile organic contaminants), measurement of the hydrogen sulfide and organic sulfur contaminant concentration, and, of course, siloxanes. Gas testing for VOCs involves obtaining a Tedlar bag or two (about 1 liter each) or metal canister (about 1 to 6 liters) of the gas. This is sent to a laboratory where the contaminants are determined by GC/MS analysis. Sampling for siloxanes is accomplished by the "Modified Dow Procedure" or methanol impinger method. (1) The siloxanes are collected by bubbling the landfill gas through impingers containing methanol. The siloxane species and their concentrations are determined by specialized GC/MS analysis of the methanol.

The results of a comprehensive LFG analysis, when correctly interpreted, provide the information necessary to engineer a gas treatment system encompassing some or all of the following processes:

1) Removal of hydrogen sulfide;
2) Gas chilling;
3) Removal of water vapor;
4) Removal of siloxanes;
5) Gas Compression;
6) Gas Drying;
7) Removal of halogenated organic species (low molecular weight contaminants containing bromine chlorine, and fluorine);
8) Separation of the methane from the carbon dioxide (methane content upgrade).

Pipeline gas injection and fuel cell applications require all 8 processes and sometimes additional treatment steps. Normally, three to five of the first five steps are employed in every LFG treatment application. Pilot test processes have been designed with up to 12 separate treatment steps for LFG applications. Additional steps can include cryogenic liquefaction of the separated methane for use as a liquid fuel and/or liquefaction of the carbon dioxide for commercial or industrial use.

Comprehensive LFG Gas Analyses

Sampling LFG for VOC analysis involves the use of one or two 1-liter Tedlar bags or a metal gas canister. The bags are filled by the motive pressure in the LFG or by use of a hand pump. These are sent to the laboratory where they are analyzed by either direct injection into a GC/MS or introduced into the GC/MS through a trap. (1, 4, 5, 6)

Since beginning testing by AFT over seven years ago, around 250 different VOCs have been found in LFG. Normally, every landfill gas will contain at least 20 to 80 of these, usually commensurate with the total VOC content of the gas. (2) Initially, tests like EPA Method 14 was used exclusively. This test evolved into a Method 14A and Method 15. It is important to note that tests like EPA Method TO-14A or TO-15 detect a specific list of 62 different chemicals considered to be harmful when released to the atmosphere. Moreover, it is a sensitive test that measures in ppbv. Some LFG contaminants have been found at levels over 100 ppmv, which is 100,000 times higher than 1 ppbv. While this test is useful for environmental reasons, it has limited applicability in total LFGQM. In order to obtain a more realistic picture of the full contaminant profile, it was necessary to develop a new test method. The new test method, called a Total VOC Survey (or TVOCS), can detect, identify, and measure the concentration to 50 ppbv to 250 ppmv of all VOCs in the biogas. While it may not be necessary to remove all of the detected VOCs from the biogas to meet a particular gas specification, it is important to know what these contaminants are and their concentrations because they can impact the overall performance of the gas treatment process and can impact emissions. Below is a partial list of landfill VOCs:

cis 1,2-Dichloroethane	1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane	1,1-Dichloroethane	1,1-Dichloroethene
1,2,4-Trichlorobenzene	1,2,4-Trimethyl Benzene	1,2-Dibromoethane
1,2-Dichlorobenzene	1,2-Dichloroethane	1,2-Dichloropropane
1,3,5-Trimethyl Benzene	1,3-Butadiene	1,3-Dichlorobenzene
1,3-Oxathiolane	1,4-Dichlorobenzene	1-Propanol
2,2,3-Trimethyl Butane	2,5-Dimethyl Thiophene	2,6-Dimethyl Heptane
2-Butanone (MEK)	2-Chloroethyl Vinyl Ether	2-Ethyl Furan
2-Ethyl Thiophene	2-Hexanone	2-Pentanone
2-Propanol	3-Butenoic Acid	3-Carene
3-Methyl Nonane	3-Methyl Thiophene	3-Methyl, 2-Butanone
4-Ethyltoluene	4-Methyl-2-Pentanone	Acetone
Acetonitrile	Alkyl Butenes	Alpha-Pinene
a-Pinene	Benzene	Benzyl Chloride
Bromodichloromethane	Bromoform	Bromomethane
Butane, 2-methyl	Butanoic Acid, Ethyl Ester	Butanols
Butene	Butoxytrimethylsilane	Butyl Acetate
Butyl Benzene	Butyl Mercaptan	C3, C4 Alkylbenzenes
C7-C8 Cyclohexanes	C9-C10 Cyclohexanes	Camphene
Carbon Disulfide	Carbon Tetrachloride	Carbonyl Sulfide
Chlorobenzene	Chloroethane	Chloroform
Chloromethane	cis-1,2 Dichloroethene	cis-1,3-Dichloropropene
Cumene	Cycloheptane	Cyclohexane
Cyclooctane	Decaline	Decanes

Dibromochloromethane	Dimethyl Disulfide	Dimethyl Furan
Dimethyl Sulfide	Dioctyl Phthalate	d-Limonene
Dodecane	Ethanol	Ethyl Acetate
Ethyl Acetate	Ethyl Butanoate	Ethyl Chloride
Ethyl Cyclohexane	Ethyl Furan	Ethyl Mercaptan
Ethyl Methyl Sulfide	Ethyl Propanoate	Ethylbenzene
Ethylene Dibromide	Freon 11	Freon 112
Freon 113	Freon 114	Freon 12
Freon 141b	Freon 142	Freon 151a
Freon 21	Freon 22	Freon 31
Furan	Heptane	Heptanone
Heptene	Hexachlorobutadiene	Hexachlorocyclopentadiene
Hexane	Hexanols	Hexanone
Hexene	Hydrogen Sulfide	Isobutane
Isopentane	Isopropyl Mercaptan	Isopropyl Methyl Sulfide
Methoxymethylsilane	Methoxytrimethylsilane	Methyl Acetate
Methyl Acetate	Methyl Bromide	Methyl Butanoate
Methyl Cyclohexane	Methyl Decaline	Methyl Ethyl Ketone
Methyl Furan	Methyl Isopropyl Ketone	Methyl Mercaptan
Methyl Propyl Ketone	Methyl Propyl Sulfide	Methyl, Ethyl Furan
Methylene Chloride	Methylpropyl Sulfide	MIBK (4-methyl, 2-pent)
m-Menthane	Monoterpenes	Naphthalene
Nonanes	n-Propyl Mercaptan	O+M+P Xylenes
Octane	Octane, 2,4,6-trimethyl	Pentamethyldisiloxane
Pentane	Pentanols	Pentyl Mercaptan
Propanols	Propyl Acetate	Propyl Mercaptan
Propylbenzene	sec-Butyl Mercaptan	Siloxane D3
Siloxane D4	Siloxane D5	Siloxane D6
Siloxane MDM	Siloxane MM	Siloxane MM
Styrene	tert-Butyl Mercaptan	Tetrachloroethene
Tetradecane	Tetrahydrofuran	Tetrahydrothiophene
Tetramethyl Stannane	Tetramethylsilane	Thiophene
Toluene	trans-1,2-Dichloroethene	trans-1,3-Dichloropropene
Trichloroethene	Trichloroethylene	Tridecane
Trimethyl Arsine	Trimethyl Bismuth	Trimethyl Stibine
Trimethylfluorosilane	Trimethylpropoxysilane	Trimethylsilanol
Undecanes	Vinyl Acetate	Vinyl Chloride

Figure 1 – Partial list of VOCs found in LFG

An inspection of Figure 1 reveals that there are 183 species and species groups listed. Including isomers, there are over 250 individually identifiable VOCs and the list is growing. This is why the information from the EPA TO14A Method is inadequate.

Part of the TVOCS is measurement of the major gas components; methane, carbon dioxide, nitrogen and oxygen. The total VOC content is also reported as "NMOC (non-methane organic contaminants) as hexane." This provides a check against the VOCs identified and quantified to determine if there is a mass balance. An estimate of the water content of the gas can be calculated from this test by mass balance, as well.

The comprehensive analysis of the biogas contaminants also reveals hydrogen sulfide and organic sulfur species such as the mercaptans, sulfides and disulfides. Most generator engine manufacturers limit the hydrogen sulfide level in the fuel gas to around 100 ppmv or even lower, in some cases. High sulfur-containing VOC contaminant levels in the LFG can result in sulfitic corrosion and in SO_x emissions to the atmosphere.

A particularly problematic group of VOCs is the halogenated compounds. In Figure 1, a little over 27% of the list is VOCs containing chlorine, bromine, and fluorine, the primary halogens associated with LFG. While these are usually present in low concentrations, they can contribute to air emissions and are very detrimental to exhaust catalysts and methane reformers.

It must be pointed out that biogas properties can vary considerably due to a number of factors: seasonal temperature changes, rainfall, gas withdrawal volume vs. landfill gas production volume, and pulling from different landfill cells. A complete VOC profile is required to assess the impact of any or all of these factors. Otherwise, the gas conditioning system may or may not function properly all of the time. Regardless of how the landfill is managed, the produced LFG should have a complete VOC profile at least once a year to determine its characteristics. The percent methane, CO_2, Oxygen, and Nitrogen is critical to the design of gas conditioning equipment which serves as pretreatment to the siloxane removal equipment and other contaminant removal equipment that may be downstream in the entire process. It is also critical to the optimum performance of generator engines and maintenance of air emission levels.

The next group of VOCs addressed during a comprehensive LFG analysis is the siloxanes. The siloxanes are determined by the SIL-1™ sampling method (methanol impinger approach) and then by direct injection of the methanol into a GC/MS for species identification and quantification. In the last paper presented at SWANA, the siloxanes and their detection limits were reported (Tower, 2003). Since then, another siloxane species has been added to the test. Previously, only cyclical siloxanes up through decamethylcyclopentasiloxane (or "D5") were reported at a detection limit of 0.3 mg/m^3. Now, dodecamethylcyclohexasiloxane (or "D6") has been added to this list. (1) Below is a table showing the detection limits of the siloxanes normally encountered in landfill gas:

Siloxane Species	Detection Limit, µg /L	Detection Limit ppb v/v
Pentamethyldisiloxane	0.3 µg /L	48.78
Hexamethyldisiloxane (MM)	0.3 µg /L	44.58
Octamethyltrisiloxane (MDM)	0.3 µg /L	30.61
Octamethylcyclotetrasiloxane (D4)	0.3 µg /L	24.41
Decamethylcyclopentasiloxane (D5)	0.3 µg /L	19.53
Dodecamethylcyclohexasiloxane (D6)*	0.3 µg /L	16.27

Figure 2 – Siloxane Species in Landfill gas and their detection limits using the SIL-1™ Method

Damage inflicted by siloxanes can be profound, causing more frequent maintenance, and lowering power generation capacity. During combustion of the LFG, siloxanes are oxidized to SiO_2, or silicon dioxide, the main ingredient in sand and glass. Analysis of deposits from engines burning LFG containing siloxanes show that additional reactions occur to form silicates, or chemicals containing the SiO_3^- group and more complex silicates. Internal combustion engine deposits are mainly silicates. Silicates contain alkaline earth metals such as sodium, potassium, magnesium, calcium and aluminum or iron or manganese, silicon and oxygen. Sometimes sulfur is also contained in these deposits. The lower molecular weight silicates are believed to be "glassy" in nature and partially molten, or "sticky" at combustion temperatures. The higher molecular weight silicates are more complex and are similar to minerals like quartz and garnet. The chemical formula for Grossular, which is found in both quartz and garnet $Ca_3Al_2(SiO_4)_3$. One deposit analyzed contained material similar to Grossular based on an approximate mass balance of the constituents.

Of course, additional chemical analyses are necessary to confirm that the material is actually Grossular, but it is important to note that the calcium and aluminum are not VOCs and are not detected in any of the gas analyses. A test method is under development to determine inorganic species in the biogas. Analysis for them is difficult because they are not volatile. Inspection of Figure 1 shows that organometallics like tin and bismuth, and organic arsenic have been detected in the VOC fraction of LFG. These chemicals were detected because they are volatile. Analysis of the condensate from LFG is one possibility for determining soluble metals, but it does not provide for a true understanding of what is in the gas, at what levels they are in the gas, and why it is possible for them to be in the gas.

Interpretation of Comprehensive LFG Gas Analyses

Once the Comprehensive LFG Gas Analysis is completed, it is interpreted by computerized modeling. The computer model determines what types of gas treatment are required based on the analytical data from the comprehensive analysis, the flow, pressure and temperature and the required gas specifications. Questions like these are answered quickly: Is hydrogen sulfide removal necessary? Is chilling the gas necessary? Is moisture removal necessary? Is siloxane removal necessary? (Usually, this is a "Yes"). Is removal of low molecular weight halogens necessary?

The ultimate SAG™ or SAGPack™ System is designed and unit processes are determined and sized during the computerized modeling. If the gas does not mass balance during modeling, approximations are made to complete the 40,000 to 50,000 individual calculations performed by the model. Some of the data the SIL-1™ computerized model provides are:

- Complete System Component Sizing
- Where consumable media is used, the predicted media life in days
- The "breakthrough" order of the VOCs in the landfill gas stream
- Pressure drop through each individual process and through the entire system
- Calculation of the mass of each individual VOC per hour, day and year that passes through the system
- Generation of "trigger" indexes (ratios of one type of VOCs to another for pointing out potential problems)
- Generation of a "siloxane fouling potential" index (measure of potential severity of silicate damage)

The output of the computerized model for a SAG™ Siloxane Removal System is summarized into one page, an example of which appears below in Figure 2 (modified to fit page). Normally, several equipment options are offered to accommodate special height and or space requirements.

Factors to Consider for Designing LFG Treatment

Another important factor to consider when evaluating the overall LFG gas treatment strategy is the power consumption by fans, compressors and chillers. Chilling below a certain point does not usually make sense from a cost/benefit perspective. For example, chilling to remove moisture can also remove from 10% to 60% of the siloxanes, depending on the species present and other gas constituents. Chilling to temperatures much below 38 ° F. (3 to 4 °C) produces diminishing returns. At lower temperatures, a little more of the siloxanes might be removed, but some of methane can become hydrated and lost with the condensate. Additionally, with chillers that operate below 32 °F (0 °C), the coalescers routinely freeze up and have to be thawed. For this reason, redundant chillers are installed and cycle back and forth to maintain adequate performance through these freeze-thaw campaigns. The parasitic load of power required to chill the gas below 30 °F, installing redundant chillers, de-icing, and potential lost methane usually does not justify low-temperature chilling. Because moisture can affect the removal of the siloxanes by the SAG™ Media, it is necessary to reduce it. The negative affect of water on the removal of siloxanes is negligible at 40 °F pressure dew point.

If a compressor is used, hydrogen sulfide is normally removed ahead of it to reduce the potential for corrosion and increased maintenance costs. For most landfill gases, a CIS™ Process (modified "iron sponge") process is adequate. For LFG with excessively high hydrogen sulfide levels (producing over about 350 lb. per day of hydrogen sulfide), alternate technologies are used, such as chemical scrubbing. The chemicals utilized in these scrubbers remove the hydrogen sulfide by converting it to elemental sulfur.

Once the hydrogen sulfide is removed from the gas, its moisture reduced, and the siloxanes removed, it is properly treated for most IC generator engines, turbines and microturbines. At this point, it is necessary to properly maintain the equipment to assure its ability to remove these three contaminants remains optimal. All necessary equipment for testing the gas is supplied to the client with notification prior to testing dates. All results are analyzed and reported back to the client promptly with recommendations on how to improve the performance of the process, if needed. Assistance with media change outs, maintenance, and testing are offered to the client where required.

SAG™ SIL-1™ Engineering Report

Note: Simulated Analysis

Project: **Revision Date 1/30/2004**

Analytical Results & Date

Siloxanes		mg/m3	ppmv	Gas Constituents, VOCs	Test Date
	Siloxane MM	14.2	2.14	Complete VOC Analysis	2/27/2002
2/21/2002	Siloxane D4	51.9	4.28	Major Gas Components	2/27/2002
	Siloxane D5	64.9	4.28	Major VOC Groups	2/27/2002

Sulfur Species	Date	Halides	Date	Other
Hydrogen Sulfide	2/25/2002	NIOSH Cl^-	N/A	
Complete Sulfur Species	2/25/2002	NIOSH F-	N/A	
		NIOSH Br	N/A	

GAS STREAM CONDITIONS:

Gas Avg. Molecular Weight:	27.13	Total Flow in SCFM:	130
Gas Density, lb/ft^3	0.0786	Pressure, psig:	2
ACF/lb.-Mole	344.91	Temperature in Deg. F.	77
Total Moles/Hr.	21.73		

Siloxane Fouling Potential				
Mild	Moderate	Heavy	**Severe**	Extreme

Species to be removed: Siloxanes MM, D4, D5

Treatment Objective: <100 ppbv (<1.0 mg/m^3) Total Siloxanes

Siloxane Species Contribution to Silicate Production (as SiO_2)				
Siloxane Species	MM	D4	D5	Total
Pounds per Year of Silicates	90.8	181.7	227.1	499.7
% of Total Silicates Produced	18.2%	36.4%	45.5%	100%
lb/1000 lb-moles gas per Year	0.018	0.037	0.046	0.101
lb./MM BTU per Year	0.16	0.32	0.40	0.88

Siloxane Removal			Estimated Media Life, Days		
SCFM	Vessel Diameter., ft.	Resistance in. w.c.	Siloxane D5	Siloxane D4	Siloxane MM
130	3.5	3.3	>365	345	77
130	3	4.7	>365	250	56
130	2.5	7.2	339	172	39

Figure 2 – Engineering Report Generated from AFT SIL-1™ Computer Model

So far, we have discussed the treatment necessary to remove hydrogen sulfide, moisture and siloxanes. In applications where the LFG must be purified to very low levels of contaminants, such as fuel cells, additional treatment steps are required. In order to remove the low molecular weight halogenated species, it is necessary to remove any remaining moisture in the LFG. In Figure 3 below, Vessel No. 4 contains a regenerable desiccant. After the LFG passes through the desiccant media, it is passed through a media that has an affinity for low molecular weight halogenated species. This media is also polymorphous graphite based. The halogenated VOC removal media is capable of reducing most of the halogenated VOCs to below 20 ppbv or lower.

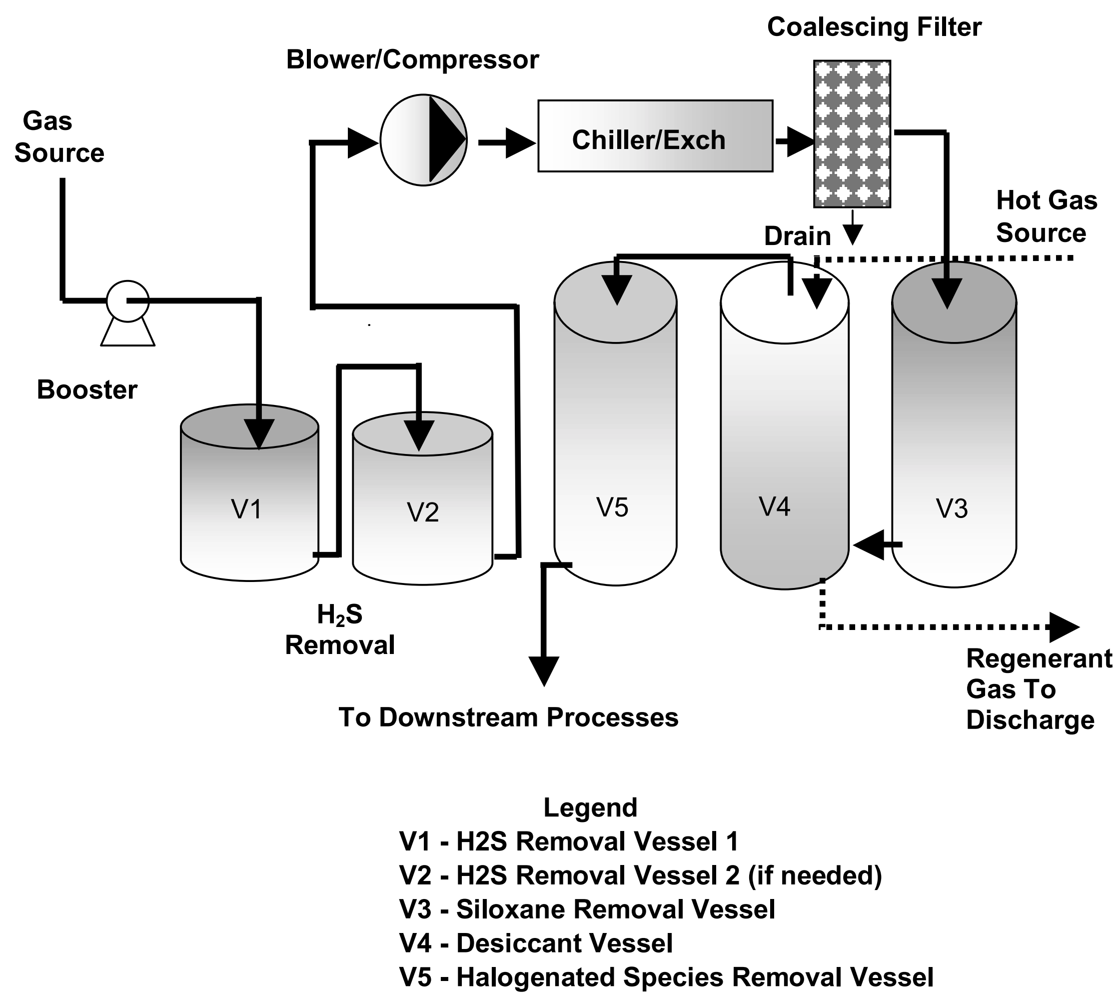

Figure 3 – SAGPack™ Process for Fuel Cell Application

Other considerations that must be made are for the correct order of the treatment components in the LFG treatment process. Earlier, we discussed the use of hydrogen sulfide removal equipment ahead of the compressor, chiller and coalescer. Another factor included in the engineering of SAGPack™ Systems is the placement of the SAG™ Siloxane Removal System. Due to economies of scale, it is often located downstream of the compressor, but there are circumstances that require it to be used on the low pressure side of the process. The factors involving the use of the SAG™ Siloxane Removal System ahead of compression must be carefully evaluated for proper system performance.

In the robust design of a LFG treatment system, the sequence of the processes is critical. Each preceding step affects the one downstream. As contaminants are removed, the LFG characteristics change and these changes must be factored into the overall treatment strategy. To better illustrate the impact of LFG constituents, the table below contains summary of their impacts:

Constituent or Contaminant	Problems Areas that Require Attention in LFG Treatment System Design and Operation				
Nitrogen	Corrosion	Can Contribute to NO_X emissions	Has to be removed for pipeline injection		
CO_2	Corrosion	Hampers Removal of some VOCs	Has to be removed for pipeline injection		
Water	Aids in Corrosion	Inhibits removal of some siloxanes and other VOCs	Enables silicate formation with siloxanes	Has to be reduced for pipeline injection	
H_2S, Sulfides	Corrosion	Can inhibit removal of siloxanes and halogenated VOCs	Produces SO_X Emissions	Have to be removed for pipeline injection	
Siloxanes	Silicate Formation	Direct impact on Equipment O&M costs	Direct impact on power production	Impact on emissions, poison to SCR, OCR catalysts	Have to be removed for pipeline injection
Oxygenated VOCs	Inhibit removal of some siloxanes	Inhibit removal of some halogenated VOCs	Contaminates condensate	Produce Odors	
Halogenated VOCs	Corrosion Potential	Contaminates condensate	Contributes to emissions	Methane Reformer Poison	

Figure 4 – Problems Requiring Inclusion in LFG Treatment System Design and Operation

Conclusion

As discussed above, when considering LFG treatment to remove siloxanes and other contaminants, the total LFGQM approach goes beyond cursory testing and installing a few processes for treating the gas. AFT has gained experience in the analysis of hundreds of LGF gas samples, the engineering and design of over 100 LFG treatment systems, and the manufacturing, installation, operation, troubleshooting, ongoing technical service, optimization, system maintenance and testing for 51 installations in the US, Canada, Europe and Pacific Rim. Total LFGQM is the only way to have a trouble-free system working around the clock to effectively and economically produce LFG that is on-specification for IC Engine Generators, Turbines, Microturbines, Boilers, Reformers, and Emission Catalysts. These steps will assure Total LFGQM:

- Proper testing of the LFG (AFT TVOCS + SIL-1™ Siloxane Analysis);
- Proper interpretation of the test results (AFT SIL-1™ Engineering Report);
- Carefully evaluating the cost/benefit of treating the gas (input by AFT);
- Inclusion of all necessary treatment processes (AFT SAGPack™ System);
- Proper sizing and design of treatment processes (SIL-1™ Computerized Modeling);
- Selection of proper materials of construction to meet gas characteristics (AFT Expertise);
- Proper sequencing of necessary treatment processes (SIL-1™ Computerized Modeling);
- Ongoing LFG influent and effluent testing to assure performance (AFT TVOCS + SIL-1™ Test Kit).

Whether you want to utilize your biogas in a boiler, IC generator engine, turbine, microturbine, inject it into a pipeline, use it as a liquid fuel, or in a fuel cell, following all of the steps in Total LFGQM will produce a successful operation.

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