Detection of Siloxane in Biogas

Developing an instrument to measure siloxanes, which are contaminants in biogas that damage gas engines

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Written by: David Ward, Safe Training Systems Ltd

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Executive summary

Siloxanes are contaminants in biogas which damage gas engines used to generate electricity. This study has investigated the feasibility of building a simple instrument that will quantify this contaminant, but will be of sufficiently low cost that it can be installed on site, thereby avoiding the current approach of sampling the gas into bags and sending them offsite to labs. A prototype instrument has been built and tested and has performed to expectations, in that we have measured standard siloxanes down to 1ppm.

Further market research continues to indicate that there is a considerable market for this instrument. A cost benefit analysis has shown that there is a considerable benefit to organisations who install this technology and who therefore protect their engines. A market size and selling price estimate indicates that an excellent commercial opportunity will be presented by the further development and marketing of this instrument.
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Glossary

Biogas Gas resulting from the biological degradation of organic matter such as sewage sludge or landfill materials, which can be used as a fuel.

FTIR Fourier Transform Infra Red – a very sophisticated technique for obtaining the spectra and hence chemical identity of organic compounds.

GCMS Gas Chromatography Mass Spectrometry – a technique where organic molecules are separated by GC and then the resulting separated molecules are identified and measured by MS.

IR Infrared radiation.

Permeation The passage of a gas or liquid through a solid material.

Siloxanes Chemicals containing carbon, hydrogen, oxygen and silicon which when burnt in engines form abrasive deposits. An example is L2, hexamethyl disiloxane.

Acknowledgements

The management of Safe Training Systems Ltd would like to thank WRAP for the support and encouragement we have received in the course of this project.
1.0 Introduction and background

1.1 The problem addressed

The problem that this study addresses is the low cost, quantitative measurement of siloxanes – a class of engine damaging gases – found in most biogases. We have identified major costs that are incurred by engine operators that could be avoided if they had better understanding of their engines’ fuels and therefore performance. A Cost/Benefit study in section 5 of this report demonstrates the savings that may be available to engine operators.

1.2 Objectives

The objectives of this feasibility study are to confirm that a market exists for such a device; produce a prototype instrument capable of detecting siloxanes at the level of interest; and to start to plan the further work needed to move the prototype design into manufacture.

1.3 Technology introduction

1.3.1 Current Technology

The current techniques for determining siloxanes in biogas use either FTIR (infrared) or GCMS (mass spectroscopy) – both techniques are extremely expensive and unsuited to field installation. Samples taken at the engine have to be transferred to a laboratory for analysis with a considerable chance that they will degrade in transfer. We aim to use an IR technique that is simpler and much lower cost than the current techniques, but will achieve similar detection levels in situ.

1.3.2 Status at the end of the contract

Reading University study All the necessary investigation of the IR technique, chemical characteristics of the family of siloxanes and the components of typical biogases have been studied and an approach developed for the instrument and the measurement.

Work at Reading University has demonstrated that real biogas samples (or at least the ones measured) have low backgrounds of potentially interfering compounds at the wavelength we wish to operate to determine the siloxanes, indicating that this technique may not suffer adversely from interference.

The Prototype A prototype has been assembled in an industry standard 19” rack and has been satisfactorily tested.

This prototype contains two decks – the upper contains the electronics, the lower the pneumatics. The electronics consist of controllers for two ovens, a power supply for the IR source, a detector control and processing board for the pyroelectric detector. Control of the instrument is by a program running on a laptop and linked to the instrument by a USB connector.

The pneumatics consist of two pumps and controllers which ensure that a steady gas flow is provided, three flowmeters, 5 solenoid operated valves, the gas cell and the Permeation tube oven. Test results show excellent performance at this stage and are reported in Section 3, Evaluation.
Market Research  Further market research has confirmed that the instrument we propose will be welcomed by the industry and that our proposed selling price of £18K will not deter the market.

1.3.3 Selection decision

The IR technology is highly appropriate – it is recognised as a standard method in gas analysis, optical components are low cost, the assemblies to mount them are simple engineered components and no vast computing power is needed to produce the analytical results.

Our final design has incorporated the IR approach described above, which we can show works well for this analytical requirement.

This is derived from the results shown in Section 3: Evaluation.

1.4 Programme outcomes

The ‘Driving Innovation’ programme is concerned with creating a larger, more efficient AD industry and within the Biogas sector, damage to engines by siloxanes is a significant cost consideration, both in downtime leading to no electrical generation and in engine repair. Better monitoring and hence control of siloxanes must therefore produce improved performance in this industry and will reduce the need for expensive engine repair.

As a microSME, the benefit to Safe Training Systems Ltd will be the opportunity to move into this new technical area, where we can already see sales volume in the UK and overseas. An environmental benefit will come from a reduction in waste – engine components, oil etc – arising from remedial actions on engines following failure.

1.5 Company overview

Safe Training Systems Ltd is a microSME specialising in scientific instrument development, manufacture and sales. We have developed products in the ionising radiation simulation, water analysis and security sectors and sell these in the UK, Europe, US and Australia. Our core technologies are microwaves, gas sensors, UV fluorescence, chemistry, physics and spectroscopy.
2.0 Technical appraisal of the technology and Phase 1 methodology

2.1 Measurement mechanics

**The Basics** Infra Red (IR) absorption is a technique that is very well established for gas analysis. Light from an IR source – generally a hot wire – is passed through a tube and is measured at the far end. Windows that are transparent to IR – such as barium fluoride – contain the gas within the tube, with an arrangement of side pipes to allow the gas which is to be analysed to enter and exit. An optical filter installed at one end of the tube – usually called a cell – selects a wavelength at which the measurement is to be made and this is specific for the gas under consideration. With only clean air in the tube, the detector reads a maximum signal, but as the concentration of the gas to be measured increases, so the IR light is absorbed by the molecules and the detector sees a diminished signal. Thus a gas can be detected and quantified.

**IR Spectrophotometers** Infrared spectroscopy has been used in analytical instruments for 80+ years and is therefore very well established. Many laboratory type instruments of excellent performance and sensitivity are sold, generally at £50k - £100k but are unsuited to field work. These instruments produce spectra allowing the measurement of many chemicals at all wavelengths. However, for an individual chemical – for instance siloxanes – measurement at one wavelength would suffice and this is the basis for our design. So a design using IR selective wavelength filters allows us to design a relatively low cost, much more rugged instrument, better suited to field work. The work to develop this prototype included considerations of the sensitivity of available IR sources and detectors and the performance of gas cells using these components. Measurements on a laboratory instrument at Reading University ensured that background contaminants in the biogas would not interfere with our measurements and that the wavelength selected was optimal for this work.

**Prototype Development** Experimental work started with 60mm lengths of aluminium tube with an IR source at one end and a pyroelectric detector at the other. Circuitry was devised to power the source and operate the detector such that both pulsed in phase, enabling the detector to operate in its designed mode.

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Figure 1 Prototype 60mm gas cell – the light path is in the upper tube
The tube – our gas cell – had barium fluoride windows at each end, which transmit IR at the required wavelength, but are not attacked by biogas. Via a side connection, gases could be passed through the tube for measurement. The two gases used at this stage were air and air with L2, a siloxane in air at a known concentration of 8000 ppm, produced by allowing the L2 to evaporate in an enclosed space, when the gas concentration could be established by reference to its vapour pressure at a known temperature.

The system easily detected this difference – the two meter shots below show the air alone signal reading 1.0220 and the air plus L2 reading 0.2433. Infrared light passing down the cell is absorbed by the siloxane, resulting in a proportionate decrease in signal.

Figure 2 Results for, left – air only and right 8000 ppm L2

While we knew that we had to achieve a 1ppm detection limit, we had three factors in hand – the length of the tube could be increased to 1000mm, better detectors were readily available and we could increase the amount of IR being emitted by the source. At this stage we needed a reliable, low level, source of siloxane standard gas in air. This was produced by a permeation tube – a short length of plugged PTFE tube, held in an oven.

The tube contained the L2 liquid and the wall thickness of the PTFE was machined so that the L2 gas would diffuse through it. Air passed over the tube carried away the gas to the cell. Varying the temperature of the oven resulted in the rate of diffusion changing in a predictable manner, so a range of concentrations between 1 and 25ppm could be achieved. Use of this device in all subsequent work enabled us to be confident regarding our measurements and it will become part of the final instrument assembly.

A picture of the permeation tube is shown below.

Figure 3 Permeation tube – note central section is made from PTFE
Further work with a 1000mm gas cell and an improved detector showed that we could increase the sensitivity sufficiently to easily achieve 1ppm, our desired target, but a 1000mm cell was physically a difficulty in instrument manufacture, so a 480mm design was assembled and found satisfactory.

Below is a picture of the completed gas cell.

**Figure 4** 480mm gas cell ready for installation in the instrument.

![480mm gas cell ready for installation in the instrument.](image)

The performance of the 480mm cell when supplied by the permeation tube is shown below – the step indicates a gas concentration of 25ppm.

**Figure 5** Performance of 480mm gas cell – measurement of 25 ppm L2

![Performance of 480mm gas cell – measurement of 25 ppm L2](image)

Biogas is a very complex mixture of methane, carbon dioxide, water vapour, sulphides and a vast number of small organic molecules. The IR technique detects most organics, but at different wavelengths dependant on their structures. We were concerned that some of these molecules might have IR absorptions at similar wavelengths to the siloxanes, so this was addressed by designing a “concentrator” which would contain an absorbent that would retain the siloxanes while allowing the other organics to pass. A concentration of the siloxanes trapped would increase the sensitivity of the instrument. Subsequent heating of the concentrator would then release the siloxanes, allowing them to be passed, free of contamination, to the gas cell.
This system only occurred to us after the WRAP Grant had been finalised and we have found alternative funding to support this.

The effect on the instrument is minimal – a small oven with a tube inside packed with absorbent – but the improvement in performance should be considerable.

The gas processing system developed consisted of a pair of pumps to regulate flow, with flowmeters so that it could be adjusted and solenoid operated switching valves to route the gas to the concentrator, gas cell or permeation tube as shown below.

**Figure 6** Diagram of gas control system

The complete instrument now consists of 5 printed circuit boards and a power supply mounted on the upper deck of a 19” rack, with the gas cell, pumps, valves and the permeation oven and concentrator oven on the lower deck.
2.1.1 Expected outcomes

We expected to find that IR was a good approach to this measurement and that components would be fairly readily available – but we were delighted to find that even with the prototype assembly, we could achieve reasonable sensitivity.

Progressing on to a built prototype has confirmed that all our electronics and pneumatics are interoperable and has resulted in an instrument of excellent performance – we can detect 1ppm of the siloxane L2.

2.1.2 Design feasibility

The feasibility of the design was checked by making up gas samples containing known concentrations of siloxanes and analysing these with the prototype assembly. This indicated that we could expect to detect 1 ppm of a common siloxane, L2. The permeation tube discussed above was also used to demonstrate long term performance over several hours. This performance has been repeated in the built prototype, attesting that our original design was satisfactory.
3.0 Evaluation including results

Reading University Results
The Chemistry Department at Reading University analysed various samples using their Perkin Elmer 7 Spectrophotometer in conjunction with the IR gas cell purchased as part of this contract.

This enabled us to investigate the IR response of various siloxanes and equally importantly, the likely interference of other compounds in biogas which might have impacted on our measurements. The results are demonstrated by the following data.

Figure 9 Graph showing the L2 gas concentration (horizontal) and the corresponding IR signal (vertical)

This graph shows the measurement of the siloxane L2 at several concentrations. Our market research indicates that this compound often occurs at 10 ppm or higher concentrations, so this graph confirms that, in the Perkin Elmer at least, we can achieve the sensitivity needed.
Detection of Siloxane in Biogas

Figure 10 Graph showing the D5 gas concentration (horizontal) and the corresponding IR signal (vertical)

\[ y = 5.76879(+/-.43157) x \]

D5 is one of the highest boiling point siloxanes and therefore may be more difficult to detect than most. Here, we can see that detection at the ppm level is easily achieved – the straight line response indicates that no adverse optical factors are influencing the results.

Figure 11 Spectra of various concentrations of D5 siloxane

This spectrum shows that, even with high BpT siloxanes, they can be satisfactorily measured in a gas cell at room temperature.
Figure 12 Two superimposed spectra – the black line is D4 in biogas, the red is added D4

The graph shows a real biogas sample analysed on the Perkin Elmer and has been recorded twice. The first record (the black one) is a sample of biogas from a landfill, taken after a filter system. The red trace is the same sample, but with 17 ppm of added D5 siloxane. This technique allows us to confirm that we are looking at the correct peak, and to make accurate estimations of the concentration of siloxanes in the biogas.

This work at Reading allowed us to have a very good insight into how analysis of biogas by the IR technique would perform, to make estimates of the response and sensitivity of the technique and to demonstrate that it actually worked on real samples.

Safe Training Systems Ltd’s On Site Siloxane Analyser

The completed prototype has been evaluated for its electronic, pneumatic and analytical performances. The electronics PCBs were tested as subassemblies before installation and were found to perform correctly as units and as a complete assembly.

The pneumatics have been pressure tested at appropriate levels and no significant leaks or malfunctions were found. Mounting the two together allowed the testing of the electronic control of the pneumatics, and these were found satisfactory.

Chemical tests using L2 were made by using the permeation tube to supply known concentrations of L2 to the gas cell and measuring the signal produced.

These results agreed with earlier work on the gas cell which was operated on pre-prototype electronics. More stability and higher responses were obtained when the instrument was tested on properly built electronics.
**Figure 13** Trace of 22 ppm siloxane L2 alternating with reference clean air being passed through the gas cell.

The upper reading is reference air, the lower is the L2.

The minimum useful reading that this instrument will detect is 20 times less than shown, so the detection limit achievable, without further instrument improvement, is 1ppm.

The fact that the trace shows no sign of short term drift or other fluctuation indicates that the system, as it now exists, is stable and is therefore an excellent basis on which to base a manufactured instrument.
4.0 Legislation¹

4.1 Applicable legislation

The only legislation of which we are aware, that will impact on our design and its installation is the ATEX (explosive atmospheres hazard) Regulations. This specifies the manufacture and testing standards needed to ensure that the product is safe to use in, in our case, potentially biogas contaminated atmospheres.

According to the Health and Safety Executive (HSE) ATEX is the name commonly given to the two European directives for controlling explosive atmospheres – ‘Directive 99/92/EC’ also known as ATEX 137 or the ATEX Workplace Directive and ‘Directive 94/9/EC’ also known as ATEX 95 or the ATEX equipment Directive. In the UK, Directive 99/92/EC is implemented by DSEAR (the Dangerous Substances and Explosive Atmospheres Regulations) 2002, whilst ‘Directive 94/9/EC’ is implemented through ‘BIS Equipment and Protective Systems Intended for use in potentially explosive atmospheres regulations 1996 (SI 1996/192)

Two approaches are possible to address this issue:

a) We can manufacture to ATEX standards
   If our siloxane monitor is to be installed in enclosures where there is a potential build-up of explosive gases, such as the iso containers which are used as engine houses, then we will need to meet ATEX standards. We have enquired about this, with the following results. This can be achieved by installing the instrument in an enclosure which is purged with nitrogen, or alternatively designing the electronics so that they are intrinsically safe. The former requires a continuing supply of nitrogen, while the latter will require much reengineering of the electronics. Cost will be a significant factor in both cases.

b) We can ensure that the monitor is installed in a safe atmosphere
   The biogas, from either a landfill or sewage treatment plant, passes from a collection system through a blower to either a filter installation or directly to the engine. Either way, there will be pipe runs between these plants where it may be possible to install our system in a safe place.

¹ At the time of writing the legislative information shown in the report is correct, but that over time it may be subject to change.
5.0 Economic/Cost Benefit Analysis

5.1 Cost Benefit Analysis

The following estimates are based on information provided by various parts of the industry. The assumption is made that Siloxane damage will begin to significantly affect engine performance within 12 months and lead to at least one major failure of the engine within 24 months requiring essential parts replacement and overhaul.

<table>
<thead>
<tr>
<th>CASE 1</th>
<th>Assuming no Siloxane Damage to Engine</th>
<th>Income</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Assuming a 1MW engine app £1m capital cost, running constantly at capacity over a 24 month period</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Generating Hours (24 months)</td>
<td>17520hrs</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Engine Generating Capacity 1000 kW/hr @ £0.05/kWhr</td>
<td>£876,000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Renewable Obligation Payment 2012-2013 @ £0.05/kWhr</td>
<td>£876,000</td>
<td></td>
</tr>
<tr>
<td><strong>Total Income</strong></td>
<td></td>
<td><strong>£1,752,000</strong></td>
<td></td>
</tr>
</tbody>
</table>

**CASE 2**

Siloxane damage requires engine to be derated at 8000hrs, and then fails at end of 17520 hr period.

<table>
<thead>
<tr>
<th>Income</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>Engine Generating Capacity 1000 kW/hr for 8000hrs @ £0.05/kWhr</td>
<td>£400,000</td>
</tr>
<tr>
<td>Renewable Obligation Payment for 8000hrs @ £0.05/kWhr</td>
<td>£400,000</td>
</tr>
<tr>
<td>Engine Generating Capacity 900 kW/hr for 9520hrs @ £0.05/kWhr</td>
<td>£428,400</td>
</tr>
<tr>
<td>Renewable Obligation Payment for 9520hrs @ £0.05/kWhr</td>
<td>£428,400</td>
</tr>
<tr>
<td>Lost income from Electricity sales</td>
<td>-£47,600</td>
</tr>
<tr>
<td>Lost Renewable Obligation Payment</td>
<td>-£47,600</td>
</tr>
<tr>
<td>Replacement Parts – Major failure (1 in 24 months)</td>
<td>-£30,000</td>
</tr>
<tr>
<td>Labour Charge 120hrs @ £50/hr</td>
<td>-£6,000</td>
</tr>
<tr>
<td>Engine Downtime 120hrs</td>
<td>-£5,400</td>
</tr>
</tbody>
</table>

**Loss of income from Siloxane Damage**  -£136,600

**Total Income**  £1,656,800

Cost of STS Siloxane Monitor  -£18,000

**Potential Saving over 2 years**  £118,600
**Customer’s viewpoint**  It would appear that, if the installation of our instrument allowed one such event to be avoided, it would more than pay for itself. If the engine was operating on particularly widely varying fuel quality, so that more than one such event a year could be avoided, then a much larger saving would result.

**Safe Training System’s viewpoint**  In the UK, there are an estimated 500 engines of this size and type, probably 3 times this many in the rest of Europe and about this combined total in the US, giving a EU/US total of 4000 engines.

If we can only make sales to customers who have suffered a failure on the scale above, and this only happens to 3% of engines, then the market is 120 units. As seems much more likely, a wider population will be proactive in protecting their plant, so perhaps 10% of operators, who may already use laboratory services to monitor their gas, may quickly become customers – giving a market size of 400 units.

STS’s best estimate of our manufacture, sales and support costs indicate a selling price of £18,000.

The market value open to this technology is then between £2.16M and £7.2M.
6.0 Commercialisation of technology

6.1 IP considerations

A single channel, non-dispersive IR – the basic technology used in this feasibility study – is used in many gas analysis instruments, including methane, carbon dioxide and ethanol breath monitors, so we cannot claim any unique position from this aspect. The measurement of siloxanes by IR is the subject of patents, but in this case the patents are written round very sophisticated, scanning, FTIR (Fourier Transform Infra Red) spectrometers and deal with the techniques used to process the IR signal. Claims are also made for the aspect of deconvoluting complex overlapping peaks, an area which does not concern us. We believe our position on the technology we are developing is that we are not infringing any existing patent, but also that everything we propose is well known in the gas analysis industry and therefore not patentable. Nevertheless, we will hold a watching brief in case a patent application position appears.

6.2 Commercialisation plans

6.2.1 Market research

We have held discussions with or visited 28 potential users so far, drawn from the landfill, sewage biogas, AD plant operators, gas purifier plant manufacturers and Analytical Laboratories.

Twenty one landfill and sewage users all stated that siloxane contamination was an increasing problem and most thought that they would need to install siloxane filters in the next few years. Fundamental to filter operation is measurement of siloxanes and the existing approaches – of gas sampling into bags or solvents, followed by transfer to a lab for analysis – was seen as inappropriate and liable to sample degradation. An installed system, with a proposed SP of £15K (more recently increased to £18K) was attractive to these user classes. Discussions held with laboratories, plant operators and engine manufacturers has indicated that the downtime costs, particularly the loss of revenue, would be a very significant support to purchase of the STS instrument.

6.2.2 Sales projection

From the Economic/Cost Benefit section in 5.1 above, we have shown a potential market size of between £2.16 and £7.2M for a product selling for £18K in Europe and the US, based on markets resulting from a small number of plant failures and a larger number of operators moving from laboratory analysis to our installed, on-site technique.

To realise these sales, we see three approaches being developed.

Within the UK, where we are generally less than 150 miles from most customers, we will sell direct to start with, then move to sales through distributors. We have two possible distributors with whom we have started outline discussions.

In Europe, we shall sell through distributors, but at this point have not held more than outline discussions with several companies who operate in this field.

In the US, we have a distributor who currently carries our water analysis instrumentation and who has expressed an interest in the siloxane instrument. His main areas of operation are in the environmental analysis sector and he is developing a particular interest in biogas analysis and processing, so may be ideal for this instrument.
As soon as we have a finished and demonstratable instrument, we will offer demonstrations to these distributors and their prime customers.

6.2.3 Manufacture

We have now identified all the components and their costs required to build this instrument, including bought-in electronic and pneumatic parts, machined parts and enclosures. From earlier work, we can estimate the cost of manufacture of PCBs and their assembly and have a clear idea of the total assembly time and therefore cost of the complete instrument.

We have not identified any part or manufacturing technique which will be difficult or unreasonably expensive to procure.

Our best estimate of the selling price for the instrument is £18k, with a sum included for distribution included and a good profit which is much in line with our earlier estimates and with the price discussed with potential purchasers.

This figure has been used in the Economic/Cost Benefit Analysis carried out in section 5.1 above.

6.2.4 Stage 2 Further developments and testing

To move from our prototype instrument to production requires several steps. A design and manufacture of professional PCBs in place of the existing prototype ones will be necessary, as will the consideration of the ATEX requirement. The current prototype is rather heavy and may not be in the best enclosure, so requiring an engineering solution, following discussions with customers.

A considerable amount of testing to confirm performance, both in handling a wide range of biogas samples and also to check that all siloxanes can be measured will be necessary. Finally and most importantly, extended reliability tests will be needed to demonstrate that the overall design is suited to routine use on landfill and sewage plant sites.
7.0 Conclusions

The key points in this study, at completion, are:

- We have assembled a prototype instrument and tested it on synthetic siloxane standards. We have achieved a detection limit for Hexamethydisiloxane (L2) of 1ppm.
- Testing at Reading University indicates that interference from other components in biogas will not be a major problem.
- Market research confirms that there is a demand and that our proposed price of £18K is acceptable.
- Cost Benefit analysis indicates that customers should welcome this product and will be able to show excellent returns on the installation.
- The likely manufacturing price and selling price of £18K give us a good profit after manufacture and use of a distributor.
- We now have the basis to move to the design of a production instrument, to demonstrate to distributors and customers.
PHASE 2 DEMONSTRATIONS

8.0 Objectives of the demonstrations

There are three objectives to this demonstration project:

8.1 Objective A

Work in the Phase 1 part of this project resulted in the design, development and assembly of an instrument that could detect the siloxane L2 down to 1 ppm, using an IR approach in both synthetic and real biogas samples. The instrument, as built, was stable, although the testing period was less than desirable.

Biogas, as produced in the landfill and sewage digester plants has a complex combination of siloxanes which varies between plants and processes and the prototype is designed to measure the sum of the siloxane components present. There are 4 L type (linear) siloxanes and 4 D type (cyclic) siloxanes to be considered.

Objective A will be the analysis of each of these chemicals in synthetic samples.

Using a correlation between IR signal, each siloxane member and total siloxanes will be derived.

8.2 Objective B

The prototype embodied all the components and designs needed in a “final” variant of the instrument, allowing us to confirm the performance and interoperability of all the parts. The working environment in which the instrument will be installed – within or very close to, the filters and engines which burn the biogas - is one requiring a specific design. This arises from the presence of explosive gases and there are strict regulations that specify how any equipment is to be built and how any explosive hazard will be controlled. The regulations involved are DSEAR (Dangerous Substances and Explosive Atmosphere Regulations) and ATEX.

Objective B will therefore involve some consultation with advisors in this area, some modification to the instrument design, then a testing phase. Testing will also be needed to meet the CE marking regulations.

8.3 Objective C

To convince potential customers that this new approach to measuring siloxanes is appropriate, we will need to demonstrate a set of test results using samples from widely differing landfill and sewage gas sites. Also, there will be a need to demonstrate long term stability.

We will achieve Objective C by two routes:

- Samples taken from many plants in the course of routine testing are available to us and we will analyse as many of these as possible and will compare our results with the laboratory’s own data.
- For the long term stability trial, we will install the siloxane monitor on a plant and data log results at 60 min intervals over a period of weeks.
Objective C will then be achieved by the generation of a data set demonstrating the accuracy, versatility and stability of the instrument.
9.0 **Methodology for demonstration**

9.1 **Delivery of demonstrations**

9.1.1 **Objective A**

Each of the siloxanes required for this study will be purchased from Aldrich or Alfa Aesar lab suppliers. The Permeation cell described in the Section 2.1 will be used to provide a steady supply of gas at a known concentration to the gas cell. Data for the set will be assembled and a comparison of response for each compound will be made. This will allow us to confirm that we can achieve equal response for each molecule and hence can calibrate the instrument for all siloxanes.

The siloxanes commonly found in biogas are shown on the table below – note that most biogas samples we have seen contain a preponderance of only 1 or 2 compounds.

<table>
<thead>
<tr>
<th>Name</th>
<th>Formula</th>
<th>Abbreviations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexamethylcyclotrisiloxane</td>
<td>C_{12}H_{18}O_{3}Si_{3}</td>
<td>D$_3$</td>
</tr>
<tr>
<td>Octamethylcyclotetrasiloxane</td>
<td>C$<em>8$H$</em>{24}$O$_4$Si$_4$</td>
<td>D$_4$</td>
</tr>
<tr>
<td>Decamethylcyclopentasiloxane</td>
<td>C$<em>{10}$H$</em>{30}$O$_5$Si$_5$</td>
<td>D$_5$</td>
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<td>D$_6$</td>
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<tr>
<td>Hexamethyldisiloxane</td>
<td>C$<em>6$H$</em>{18}$Si$_2$O</td>
<td>L$_2$, MM</td>
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<tr>
<td>Octamethyltrisiloxane</td>
<td>C$<em>8$H$</em>{24}$Si$_3$O$_2$</td>
<td>L$_3$, MDM</td>
</tr>
<tr>
<td>Decamethyltetrasiloxane</td>
<td>C$<em>{10}$H$</em>{30}$Si$_4$O$_3$</td>
<td>L$_4$, MD$_2$M</td>
</tr>
<tr>
<td>Dodecamethylpentasiloxane</td>
<td>C$<em>{12}$H$</em>{36}$Si$_5$O$_4$</td>
<td>L$_5$, MD$_3$M</td>
</tr>
</tbody>
</table>

At the completion of this objective, we will know that the instrument will successfully quantify all the siloxanes in common use and that there are no problems with interference.

9.1.2 **Objective B**

The need for ATEX and possibly DSEAR qualification appeared late in the earlier design phase and so was not considered from the start. However, there are a large number of ways of achieving compliance and these include enclosing the instrument in a sealed, purged case, or separating electronics and potential sparking parts from the area of hazard, to encapsulation or oil immersion.

The test houses who provide the certification to ATEX standard also provide advice and consultancy and we shall work along with our selected test house to improve our design to ensure that we achieve ATEX at minimal cost, without degrading our instrument performance.

We will be assembling two more instruments to the design required to meet the ATEX requirement – these will have mainly identical parts to the first and will allow us to retain the
first prototype for further testing, have one ATEX design for destructive testing and an ATEX design to demonstrate to customers, as described in Objective C below.

The ATEX instruments will be tested by the same test house for CE marking regulations.

9.1.3 Objective C

Wide range sample testing.

It will be necessary to analyse a very wide range of real biogas samples to convince ourselves and our customers that there will be no interferences preventing a successful siloxane analysis. The only way to do this is to analyse real samples – there is no alternative.

We have access to biogas samples from a test house – these will come to us without identification but with some analysis and our interest will be in measuring the siloxanes present, but also in looking for non-siloxane chemicals which may interfere with our analytical technique.

We estimate that if we could process 50 samples from separate sites, the statistics on the results would give us total confidence in this method.

This work will require an instrument to be available at short notice – because the samples may not be stable – so it will be performed on the first prototype.

The second part of Objective C is the long term stability tests. We are discussing this with one of the Water Companies who, we hope, will give us access to a plant with a biogas engine. It is necessary to show 12 weeks continuous operation to convince us - and our customers – that this is a viable instrument.

We expect to have the ATEX approved design built by this stage, so we can also consider the final installation arrangements on real sites.

9.1.4 Key project milestones

<table>
<thead>
<tr>
<th>Milestone</th>
<th>Time required</th>
<th>Time from start</th>
</tr>
</thead>
<tbody>
<tr>
<td>Milestone 1 Obj A siloxane L and D tests</td>
<td>8 weeks</td>
<td>w2-w10</td>
</tr>
<tr>
<td>Milestone 2 Obj B ATEX design</td>
<td>8 weeks</td>
<td>w4-w12</td>
</tr>
<tr>
<td>Milestone 3 Obj B ATEX build</td>
<td>6 weeks</td>
<td>w12-w18</td>
</tr>
<tr>
<td>Milestone 4 Obj B ATEX tests</td>
<td>2 week</td>
<td>w18-w19</td>
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<tr>
<td>Milestone 5 Obj C Real Biogas tests</td>
<td>8 weeks</td>
<td>w10-w18</td>
</tr>
<tr>
<td>Milestone 6 Obj C Long term stab tests</td>
<td>12 weeks</td>
<td>w20-w32</td>
</tr>
<tr>
<td>Milestone 7 Report</td>
<td>1 week</td>
<td>w33</td>
</tr>
</tbody>
</table>

9.2 Securing of sites and stakeholders

Site trials cannot start until we have built and had tested an ATEX approved instrument.
We are currently in discussion with 3 companies regarding trials of our equipment on their sites. Two of these companies supply gas filtration equipment, are very keen to see our instrument progress to market and assure us that, when we have an instrument, they will provide a site. There are, we think, two possible drawbacks with these companies. The first is that they do not actually operate the sites they are considering, only equipment on the site, so access may be more difficult than they suggest. Both have suggested that such access may be linked to their becoming our distributor, which may tie our hands beyond what is in our interests.

The third company is Thames Water Plc, who have many sites, over 20 of which have installed gas engines. David Linsell CHP Manager for Thames Water has indicated a serious interest in our developments and has suggested that Thames will find a site suitable for this trial. Being a major organisation, decisions will be slow in coming, but we hope to hear from them in a few weeks.

Clearly, the instrument would have to operate to ATEX standard and we assume that any installation costs would be for us to settle.

Other than some consultancy, there will be no stakeholders or contractors involved in this project. All work will be undertaken by STS personnel.

9.3 Project timescale

We have set out a table of Milestones at 9.1.4 and attach a project Gannt chart, we trust that it will fulfil your requirements – as far as we can.

9.3.1 See Appendix 1
Spread sheet WRAP 2 Gannt
10.0 Cost breakdown

10.1 See Appendix 2

Spread sheet WRAP 2 costs

10.2 Project financing

We have had discussions with several companies in the biogas industry regarding their partially funding the development of the siloxane monitor. While two have indicated a serious interest in our work, we think that the terms that they would impose would not be acceptable to us.

We are continuing to search for a suitable funding organisation, with whom we could comfortably operate with on this project.

Our own resources are stretched at the moment, but we think the potential for the siloxane monitor is such that we are prepared to contribute £7,500 to this development. We hope that WRAP will be able to provide the balance.

10.3 Finance secured

Our contribution to the financing of this project will come from our own reserves; hence there is no financial security consideration, nor, therefore any obligation on us.
11.0 Key personnel

The team will consist of 4 of our members of staff, selected for their specialist skills in their work area indicated.

DR Ward, Managing Director, has 45 years of experience in all aspects of the analytical chemistry business, including working for major manufacturers, test laboratories and for 25 years operating his own instrument manufacturing business.

He will be responsible for the laboratory based tests and for arranging and supervising the ATEX testing and some of the assembly of the ATEX instrument.

DC Lambourne, FCIS Financial Director, has worked in company accountancy and finance areas for 40 years. He will be responsible for controlling expenditure and preparing financial statements at the end of the project.

Christopher S Fifield, BSc, M Phys, Technical Manager, has worked with STS for 3 years on instrument design. He will undertake electronic assembly of the ATEX instrument, software writing and supervision of the on-site testing.

Jim Ward HND, has worked for STS for 4 years in marketing, procurement and sales and will carry out instrument assembly, procurement of parts and subcontracted services and also some site testing.

Two consultants will be used in this work – Nigel Collier, who will advise us on some aspects of instrument design for gas analysis, and BASEFA Ltd who will probably carry out the consultancy and testing for the ATEX approvals.
12.0  Evaluation and monitoring

12.1  Monitoring regime and methodology

The siloxane monitor has three modes of operation – measurement of siloxane in biogas; measurement of clean reference air; and finally measurement of a siloxane-in-air standard generated within the instrument.

The instrument is therefore self-checking and self-calibrating.

All the work undertaken in this second stage of the project will therefore be automatically calibrated, allowing us to have confidence in the results, to identify drift and irregular behaviour.

Evaluation will therefore follow the route described in Section 8.3 above, where the instrument will be applied to as wide a range of samples as possible, with the instrument continually self-checking its performance.

All the data generated will be stored in spread sheets and subject to statistical analysis so that we can extract the fine detail regarding the performance of the instrument.

In addition to the primary output of siloxane concentration, there will be a considerable amount of monitoring of temperatures, waveforms, signals on test points and pressure variations throughout the system and all this supporting data will be stored for performance analysis purposes.
### 13.0 Health, safety and risk

#### 13.1 Risk assessment

Risks may arise in three areas of this project – at Safe Training Systems Ltd site; travelling; and at customers’ and suppliers’ sites.

**Risks at STS**

Risks at STS occur in two main areas, electrical and explosive gases hazards.

The instrument is powered from 240V mains, but within the instrument high voltages are restricted to a small, enclosed area. Assembly of this part of the instrument will only be undertaken by suitably qualified staff.

Various small heaters in the instrument are installed in metal housings which are enclosed in insulation, so no possibility of burns occurs. Biogas samples will arrive in 10litre bags and will contain methane (c60%), carbon dioxide (c40%), ppm levels of sulphides, mercaptans, hydrocarbons and siloxanes. Release of the entire contents by rupture would place this gas in the atmosphere of the laboratory. While the methane is explosive if diluted by air to between 5 and 15%, the small sample bag volume – 10 litres - compared with the volume in an average laboratory means that no explosive hazard will be present. The carbon dioxide presents an asphyxiation hazard, but likewise dilutions will be such that no hazard will arise. The other components, while presenting bad smells, will not constitute a hazard because of their low concentrations.

The gas handling system operates at 10psi and is assembled from components rated at 100psi, so no bursting risk is present.

The prototype built in the Phase 1 part of this project weighed 22 kg but we expect the ATEX standard one to weigh slightly less. Staff will be instructed in the correct handling technique for items of this weight.

**Risks when travelling**

A considerable time will be spent on site and therefore travelling by car and train will be necessary. All staff have been instructed that they should obey all motoring regulations at all times.

**Risks on site**

We do not expect to be allowed on site unaccompanied – the landfill and sewage treatment industries are well aware of their obligations to visitors. We carry standard safety equipment for use on site, as required by the site operators and there is a company instruction that this should be used at all times.

13.1.1 *See Appendix 3 Risk Assessment & H&S Policy*

#### 13.2 Future key health and safety considerations

At this point, we cannot see any unforeseen H&S issues that could occur in this project, or subsequently – but we will hold a watching brief during the course of this work for issues that could impact on the H&S area.
Appendix 1

Safe Training Systems Ltd

Project code: 1928
ISBN: OIN001-010

Milestone 1
Task: Project Management Planning
Objective A L&D Siloxane Tests

Milestone 2
Task: Design
Objective B ATEX Instrument Design

Milestone 3
Task: PCB Manufacture
Objective B Instrument build

Milestone 4
Task: Test
Objective B Instrument test for ATEX

Milestone 5
Task: Software Writing
Objective C Real Biogas Tests

Milestone 6
Task: Test & Monitoring
Objective C Long Term site tests

Milestone 7
Task: Evaluation/Decomm
Final Report production

Time /weeks: 0 2 4 6 8 10 12 14 16 18 20 22 24 26 28 30 32 34
## Appendix 2

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<thead>
<tr>
<th>Safe Training Systems Ltd</th>
<th>WRAP 2 Costs (excluding VAT)</th>
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<td><strong>ISBN:</strong> OIN001-010</td>
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|                      |                |          |          |          | **£92,071**|

**Total**
## Appendix 3 H&S Policy & Risk Assessment

This is the statement of general policy and arrangements for:

### Overall and final responsibility for health and safety is that of:

<table>
<thead>
<tr>
<th>Safe Training Systems Ltd</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jim Ward</td>
</tr>
<tr>
<td>Jim Ward</td>
</tr>
</tbody>
</table>

### Day-to-day responsibility for ensuring this policy is put into practice is delegated to:

<table>
<thead>
<tr>
<th>Statement of general policy</th>
<th>Responsibility of (Name / Title)</th>
<th>Action / Arrangements</th>
</tr>
</thead>
<tbody>
<tr>
<td>To prevent accidents and cases of work-related ill health and provide adequate control of</td>
<td>Jim Ward Operations Manager</td>
<td>Workshop areas to be kept clean and tidy, chemicals correctly stored, trip hazards</td>
</tr>
<tr>
<td>health and safety risks arising from work activities</td>
<td></td>
<td>assessed and mitigated. Reporting of issues to JW</td>
</tr>
<tr>
<td>To provide adequate training to ensure employees are competent to do their work</td>
<td>Jim Ward Operations Manager</td>
<td>Supervised training before initiation of job, risk assessment for new procedures,</td>
</tr>
<tr>
<td></td>
<td></td>
<td>employee aware of responsibility to be vigilant.</td>
</tr>
<tr>
<td>To engage and consult with employees on day-to-day health and safety conditions and</td>
<td>Jim Ward Operations Manager</td>
<td>Act on any identified issues and work to prevent occurrence.</td>
</tr>
<tr>
<td>provide advice and supervision on occupational health</td>
<td></td>
<td></td>
</tr>
<tr>
<td>To implement emergency procedures - evacuation in case of fire or other significant</td>
<td>Jim Ward Operations Manager</td>
<td>JW to act as Fire Officer unless absent, CF to act as deputy, Emergency procedure</td>
</tr>
<tr>
<td>incident. You can find help with your fire risk assessment at: (See note 1 below)</td>
<td></td>
<td>guidance to be agreed and adhered to by all staff.</td>
</tr>
<tr>
<td>To maintain safe and healthy working conditions, provide and maintain plant, equipment</td>
<td>Jim Ward Operations Manager</td>
<td>All responsible for own work areas, issues to be reported to JW</td>
</tr>
<tr>
<td>and machinery, and ensure safe storage / use of substances</td>
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<td></td>
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<tr>
<td>Health and safety law poster is displayed:</td>
<td>In Workshop/Lab &amp; Office</td>
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<tr>
<td>First-aid box and accident book are located:</td>
<td>In Workshop</td>
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<tr>
<td>Accidents and ill health at work reported under RIDDOR: (Reporting of Injuries, Diseases and Dangerous Occurrences Regulations) (see note 2 below)</td>
<td>Recorded in Accident book if reporting required</td>
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</tbody>
</table>

| Signed: (Employer) | J.Ward | Date: | 01/03/2013 |
| Subject to review, monitoring and revision by: | D Ward | Every: | 12 months or sooner if work activity changes |
### Safe Training Systems Ltd: Siloxane Monitor Risk Assessment

<table>
<thead>
<tr>
<th>What are the hazards?</th>
<th>Who might be harmed and how?</th>
<th>What are you already doing?</th>
<th>Do you need to do anything else to manage this risk?</th>
<th>Action by whom?</th>
<th>Action by when?</th>
<th>Done</th>
</tr>
</thead>
<tbody>
<tr>
<td>Slips trips and falls</td>
<td>All Staff and visitors</td>
<td>Clean and Tidy work Environment, cables and trailing leads removed where possible and routed out of way. Obstacles removed or clearly marked</td>
<td>Monitor and resolve as required</td>
<td>ALL</td>
<td>Ongoing</td>
<td></td>
</tr>
<tr>
<td>Travel</td>
<td>All Staff</td>
<td>Vehicles used for business purposes to be maintained to expected standards, drivers to adhere to regulations as expected</td>
<td>NO</td>
<td>ALL</td>
<td>Ongoing</td>
<td></td>
</tr>
<tr>
<td>Site Visits</td>
<td>All Staff</td>
<td>Staff should not be unaccompanied on external sites, all advisory notices and legislation of the site is to be followed. Staff should attend sites with Personal Protective equipment as required. Particular attention to be paid to vehicle movements on unfamiliar sites.</td>
<td>Monitor and report any incidents for update to advice as required</td>
<td>JW</td>
<td>Ongoing</td>
<td></td>
</tr>
<tr>
<td>Lifting and Handling</td>
<td>All Staff</td>
<td>Correct handling procedures should be maintained at all times. The Siloxane Monitor weighs approx 22KG and should therefore be lifted where possible by two people. Where this is not possible lifting should be restricted to the minimum.</td>
<td>Monitor and advise on handling procedures</td>
<td>ALL</td>
<td>Ongoing</td>
<td></td>
</tr>
<tr>
<td>Electrical Hazard</td>
<td>All Staff</td>
<td>The Siloxane monitor is supplied from a 240V supply and must therefore only be operated and maintained by competent staff. Particular care must be taken when working on the internal electronics and the mains lead must be disconnected before entering the casing. Substantial shock and damage to health is likely if this is not adhered to</td>
<td>Monitor and train all users, ensure adequate labelling identifying hazard is in place and maintained, prevent untrained users from working on instrument</td>
<td>JW</td>
<td>Ongoing</td>
<td></td>
</tr>
<tr>
<td>What are the hazards?</td>
<td>Who might be harmed and how?</td>
<td>What are you already doing?</td>
<td>Do you need to do anything else to manage this risk?</td>
<td>Action by whom?</td>
<td>Action by when?</td>
<td>Done</td>
</tr>
<tr>
<td>----------------------</td>
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<tr>
<td>Explosive Hazard</td>
<td>All Staff</td>
<td>Gas sample bags contain potentially explosive gases and must be handled with care and kept away from naked flames and sources of ignition. Gas bags to be stored away from workshop in secure building. All staff handling gas bags to be conversant with appropriate handling techniques. Work areas to be kept well ventilated at all times.</td>
<td>Monitor handling procedures and ensure trained staff following guidelines</td>
<td>JW</td>
<td>Ongoing</td>
<td></td>
</tr>
<tr>
<td>Chemicals</td>
<td>All Staff</td>
<td>Chemicals to be kept in specified areas and in original containers with MSDS sheets. Handling procedures to be followed</td>
<td>Monitor and Review as required</td>
<td>DW</td>
<td>Ongoing</td>
<td></td>
</tr>
</tbody>
</table>