

Lonestar Information

Troubleshooting Contamination of Lonestar Systems



Author(s) Jonathan Angove
Date 09/12/10



Contents

Executive Summary.....	3
System Handling Overview	3
The Lonestar Platform.....	5
Lonestar Initial Installation	8
Laboratory Glassware Cleaning.....	10
Diagnosing the Site of Contamination	11
Appendix 1. Flow-chart to ensure the Lonestar system is clean on installation.....	12
Appendix 2. Flow-chart of diagnosing contamination due to a sample being analysed.....	14



Executive Summary

This document is intended to be an aid in troubleshooting contamination problems seen using a Lonestar system. This guide should be used by staff members trained in the use of the Lonestar system. Some parts of the Lonestar system are heated to high temperatures. Care should be taken to ensure that the system has cooled sufficiently before the heated parts are handled. The Lonestar system contains a radioactive ion source (Ni^{63}); under no circumstance should a Lonestar user attempt to open the Lonestar unit as this must only be performed by trained personnel in a controlled environment.

System Handling Overview

It is important to wear powder free nitrile gloves when handling any sample container, pipe work or fittings as to prevent finger grease from contaminating the Lonestar system. Ensure that the Lonestar system is correctly installed; Figure 2 shows schematics of the Lonestar system. Figure 3 and Figure 4 are photographs of a Lonestar system that has been installed and is in use. Clean, dry air is used within Owlstone for use with the Lonestar systems (dew point - 45°C, hydrocarbon content <1ppm of methane). The gas supply should be connected first to the supplied pressure regulator, then to the supplied toggle valve before being connected to the base of the supplied scrubber on the right side of the Lonestar unit. The supplied needle valve should be installed on the exhaust port from the Lonestar unit. 1/8" pipe is used to connect the clean gas outlet through the sampling device to the sample inlet filter.

The filter is heated and a 1 μm PTFE filter must be installed to ensure no particulates from the sample enter the Lonestar unit. It is worth stressing that the Lonestar unit is a vapour phase detection system. Under no circumstance should staff attempt to pass liquid samples into the Lonestar system. If liquid enters the Lonestar system, the sampling device should be isolated from the Lonestar unit by setting the Lonestar Purge ON. The supply of air should then be stopped to the Lonestar unit using the toggle valve in the gas line to the right of the Lonestar unit. The pressure will drop within the Lonestar system. A member of the Owlstone Service and Support department should then be contacted for further advice.

The Lonestar system is set up in the Owlstone Quality Assurance department to ensure the cleanliness of the system, including the filter provided. Figure 5 is a photograph of a Lonestar system in Owlstone's Quality Assurance department. Upon installation, the Lonestar system should be set up as shown in Figure 5 and the system run under the conditions detailed in Table 1. With the Gas Purge ON, blank spectra similar to that seen prior to the instrument shipping should be obtained. An example of a blank DF Matrix is shown in Figure 1.



Lonestar Parameter	Set Point
Gas quality	Air, dew point -45°C, hydrocarbon content <1ppm methane
Gas flow	1.8Lpm
Chip pressure	1.0bar _g
Configuration file	Lonestar Checkout
Field intensity Start	0%
Field intensity End	100%
Number of lines	51
Min DF Matrix Interval, sec	0
Min CV Sweep Interval, sec	0
Average CV Sweeps	1
Pump	OFF
Gas purge	OFF
Sensor Heater	ON
Filter Heater	ON
Filter temperature	>99°C
Motherboard temperature	>38°C
Sensor head temperature	>65°C

Table 1 Lonestar parameters used at Owlstone to ensure system cleanliness

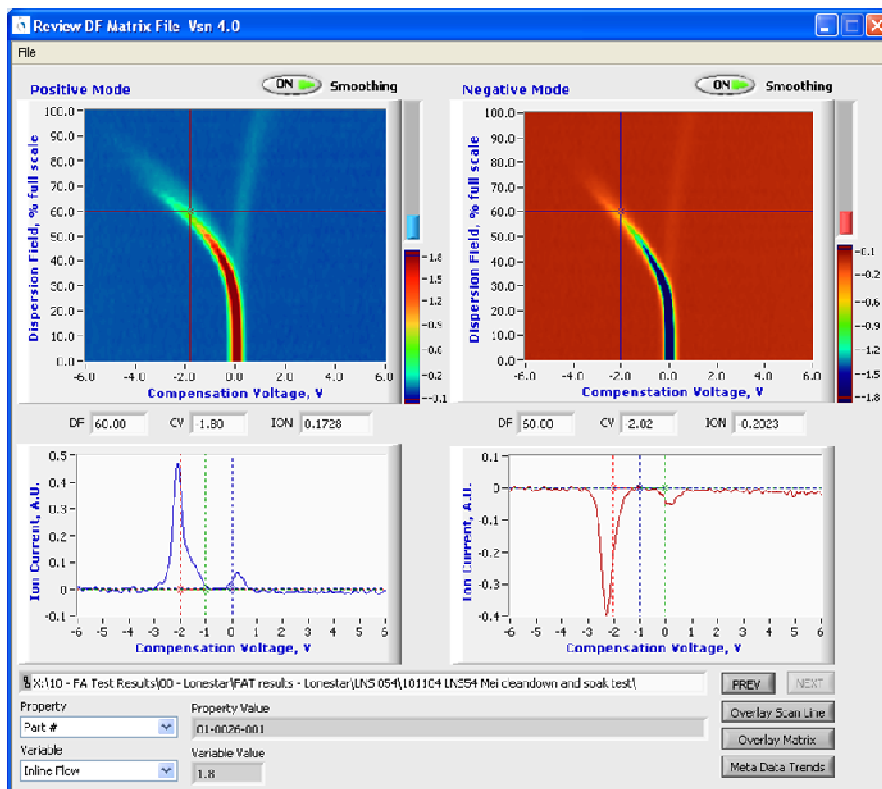


Figure 1 Example DF Matrix of a Lonestar system prior to shipping



The Lonestar Platform

Lonestar is a powerful and adaptable chemical monitor in a portable self contained unit. Incorporating Owlstone's proprietary FAIMS technology, the instrument offers the flexibility to provide rapid alerts and detailed sample analysis. It can be trained to respond to a broad range of chemical scenarios and can be easily integrated with other sensors and third party systems to provide a complete monitoring solution. As a result, Lonestar is suitable for a broad variety of applications ranging from process monitoring to lab based R&D.

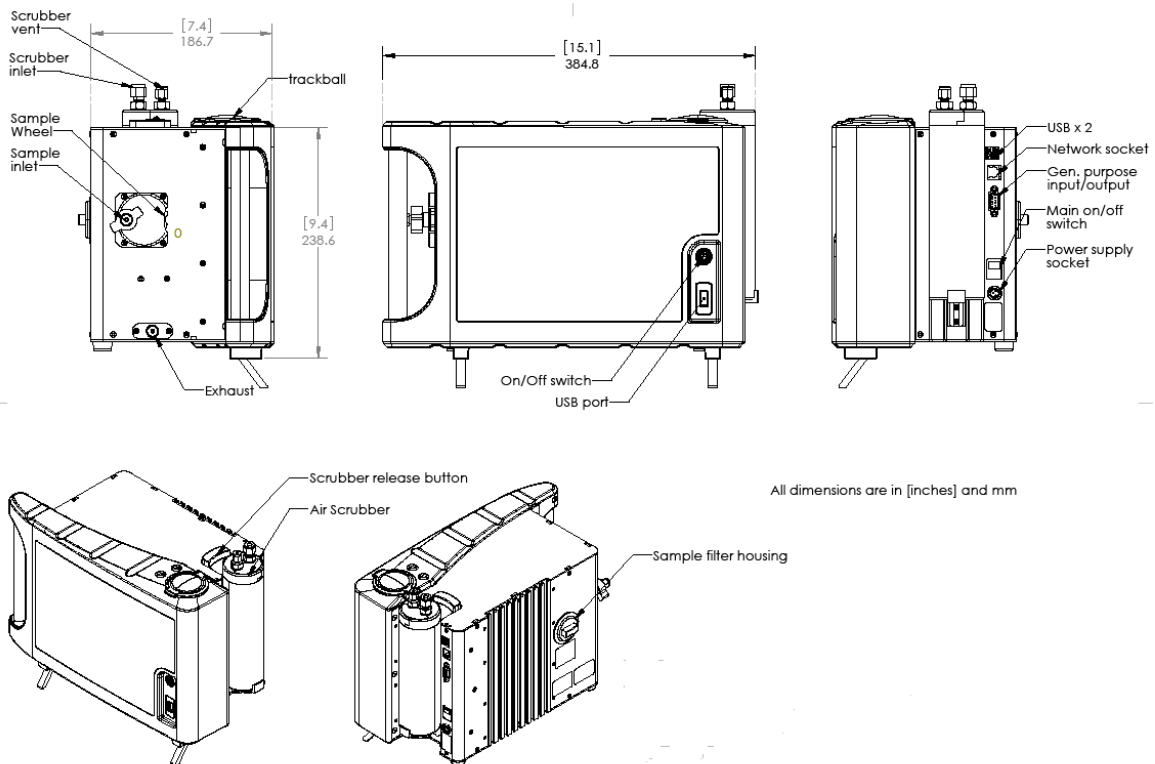


Figure 2 Lonestar connection figures

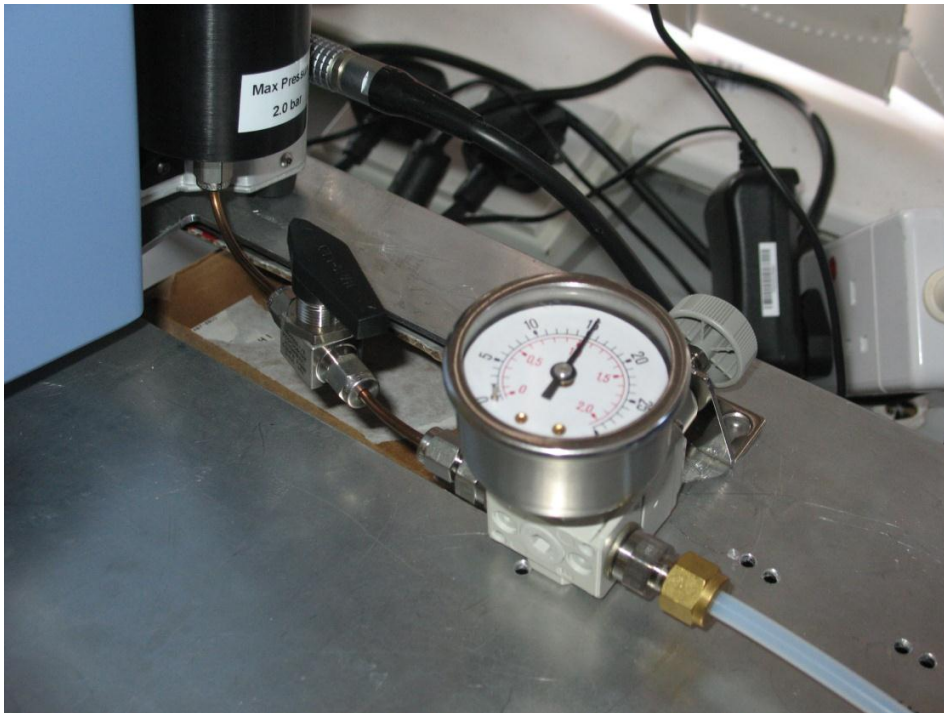


Figure 3 A photograph of the installation of the pressure regulator and toggle valve in the carrier gas flow immediately prior to the scrubber mounted on the right side of the Lonestar unit

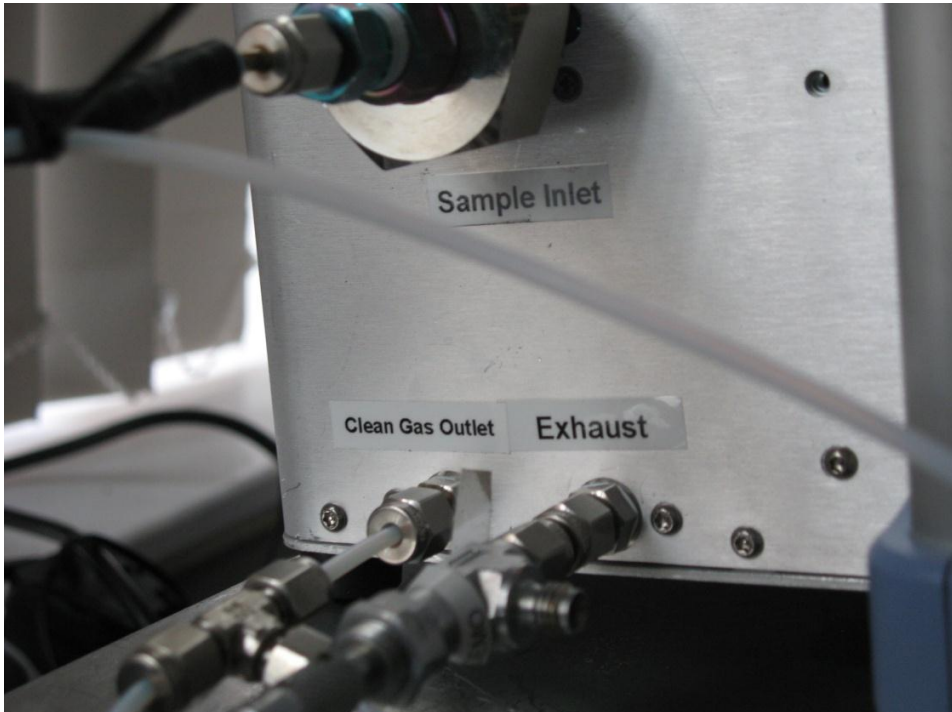


Figure 4 A photograph of the installation of the needle valve on the exhaust port to provide a restriction to control the pressure at the chip



Figure 5 A photograph of a Lonestar unit set up within the Owlstone Quality Assurance department. Note the needle valve installed in the exhaust port and the loop of 1/8" PTFE pipe connecting the clean gas outlet with the filter mounted in the sample inlet. The filter tested within the Quality Assurance department is shipped with the Lonestar unit



Lonestar Initial Installation

When a Lonestar system is first installed, it should be installed as shown in Figure 3, Figure 4 and Figure 5. The Lonestar Checkout configuration file should be loaded, and the conditions detailed in Table 1 used.

Connect the Clean Gas Outlet to the filter as shown in Figure 5. Collect data, but keep the Gas Purge ON. With the Gas Purge ON, the carrier gas passes through the pressure regulator and toggle valve before being further cleaned by the Lonestar scrubber. The clean carrier gas is diverted by a solenoid valve within the Lonestar system, isolating the flow from the sample introduction module, for ionisation and detection by the Lonestar. Press the START button, collecting the data if desired, and allow the system 15 minutes for all the temperature setpoints within the Lonestar system to be reached.

After this time, a blank DF Matrix similar to that shown in Figure 1 should be obtained. If 10 further DF Matrices are collected and there is contamination in each of the DF Matrices, then the Lonestar is contaminated. Ensure that the Lonestar system has been correctly installed, with the carrier gas passing through the scrubber.

If the system has been correctly installed, but the contamination is still seen, examine the data critically. Is the peak reducing in Ion Current intensity from DF Matrix to subsequent DF Matrix? If so, the system is slowly cleaning down. If not, contact the Owlstone Service and Support department for further advice.

If the system shows a clean DF Matrix, similar to that obtained at Owlstone, then the Gas Purge should be set to OFF, and further DF Matrices collected. By setting the Gas Purge to OFF, the carrier gas is diverted by the Lonestar solenoid valve through the sampling device and then into the Lonestar system for analysis. If any contaminant peaks are now seen, they are from the pipe connecting the Clean Gas Outlet to the filter, or from the filter itself. If 10 DF Matrices are collected and all of them now show the contaminant peak, ensure that the Filter Heater has been set ON, and it has reached a temperature of 100°C. If this is the case, examine the data critically. Is the peak reducing in Ion Current intensity from DF Matrix to subsequent DF Matrix? If so, the system is slowly cleaning down. If not, replace the pipe connecting the Clean Gas Outlet to the filter, ensuring that nitrile gloves are worn. If the system continues to show contamination, then the filter should be replaced; ensuring that the Filter Heater is turned OFF and the filter has cooled. If the system continues to show contamination peaks, contact the Owlstone Service and Support department for further advice.

Once the Lonestar system has been shown to be clean after delivery, then install the desired sample introduction module, ensuring that nitrile gloves are worn. Allow sufficient time for the sample introduction module to achieve the desired temperature set point. Collect further DF Matrices with the Gas Purge OFF, so the carrier gas is being flushed through the sample introduction module. If contamination is now seen, it is coming from the sample introduction module. If the sample bottle is made of glass, ensure it has been cleaned as detailed later in



this procedure, titled “Laboratory Glassware Cleaning”. If the sample bottle is made of plastic, ensure that a sample temperature below 50°C is being used as contaminant peaks may be out-gassed from plastics above this temperature.

If 10 DF Matrices are collected and they all show contamination, examine the data critically. Is the peak reducing in Ion Current intensity from DF Matrix to subsequent DF Matrix? If so, the system is slowly cleaning down. If not, replace the sample bottle, ensuring that nitrile gloves are being worn and that the bottle is clean. Is the contaminant peak still present, or has it changed, or reduced significantly in Ion Current response? If it is still present, then the contamination is not from the sample bottle. If the contaminant peak has been replaced with a different contaminant peak, then both sample bottles are contaminated and should be replaced by clean ones. If the Ion Current response of the contaminant peak has been significantly reduced, allow further DF Matrices to be collected to see that the contaminant peak is no-longer detected.

If the entire system is now clean, then samples may be analysed.

Appendix 1. Flow-chart to ensure the Lonestar system is clean on installation is a flow-chart summarising the procedures detailed in this section of the procedure.



Laboratory Glassware Cleaning

If glassware has been used, dispose of the sample using the appropriate waste disposal method. Rinse the glassware with deionised water (Millipore, conductivity 18.2 $\text{M}\Omega \cdot \text{cm}$ @ 25°C) and dispose of this appropriately. Rinse the glassware with a solvent that is both water miscible and able to dissolve the analyte and, or matrix being used (ethanol or acetone are typically used). Dispose of this solvent appropriately.

Place the glassware in a laboratory glassware washer capable of using both a detergent wash and an acid rinse. Wash the glassware and place in a drying cabinet at a temperature above 60°C and allow to dry. This stage is also useful in removing any moderately volatile contaminants on the glassware. Glassware should be stored in the cabinet for at least an hour before use.

It is possible, if glassware has become heavily contaminated, that one cycle of the dishwasher is not sufficient to clean the glassware. If it is suspected that this will be the case, then one piece of glassware should be taken from the dishwasher and analysed as a blank on the Lonestar unit before any further glassware is removed from the dishwasher. If the glassware is blank, then the dishwasher can safely be emptied into the drying cabinet. If not, then the glassware should be re-washed and tested until it is clean. By ensuring a sample of the glassware is clean before placing the entire contents of the dishwasher in the drying cabinet you ensure that you do not contaminate the rest of the contents of the drying cabinet.



Diagnosing the Site of Contamination

If a high-concentration sample is analysed, then parts of the Lonestar system can become contaminated. Replace the sample bottle with an empty, clean, dry sample bottle. With the Gas Purge ON, collect data. If analyte or matrix peaks are detected, then these are residual analyte/matrix within the Lonestar system. Allow the Lonestar system sufficient time for a clean DF Matrix to be obtained, as shown in Figure 1.

Once the system is clean, set the Gas Purge OFF and collect further data. If further matrix/analyte peaks are seen, these are carry-over from the previous sample. If new contaminant peaks are seen, then these are from the new glassware. Typical sites of contamination are the lid of the sample bottle, the pipe from the sample bottle to the Lonestar filter, any unions used in the sample flowpath or the filter.

A systematic approach to diagnosing the site of contamination is required so as not to mislead the analyst. You must begin the troubleshooting process closest to the sample, working towards the Lonestar system.

We have established that the Lonestar system is clean from the Gas Purge ON data and have replaced the sample bottle. If the analyte/matrix contamination is still present, then the lid of the sample bottle should be replaced next with a clean one. If the contaminant peaks are significantly reduced, allow the system time to clean down prior to the subsequent sample. If not, then the pipe from the sample bottle should be replaced next. If the contaminant peaks are significantly reduced, allow the system time to clean down prior to the subsequent sample. If not, then the next part of the sample flowpath should be replaced until either the contaminant peaks have been isolated and removed, or the filter has been replaced.

If the filter has been replaced, but the contaminant peaks are still present, set the Gas Purge ON once more and ensure that the Lonestar system is clean. If all the sample flowpath has been replaced, but the contaminant peaks are still present, then the filter body or the PTFE discs in the filter are contaminated. If this is the case, please contact a member of the Owlstone Service and Support department for information regarding further heating the filter or alternate sample introduction modules.

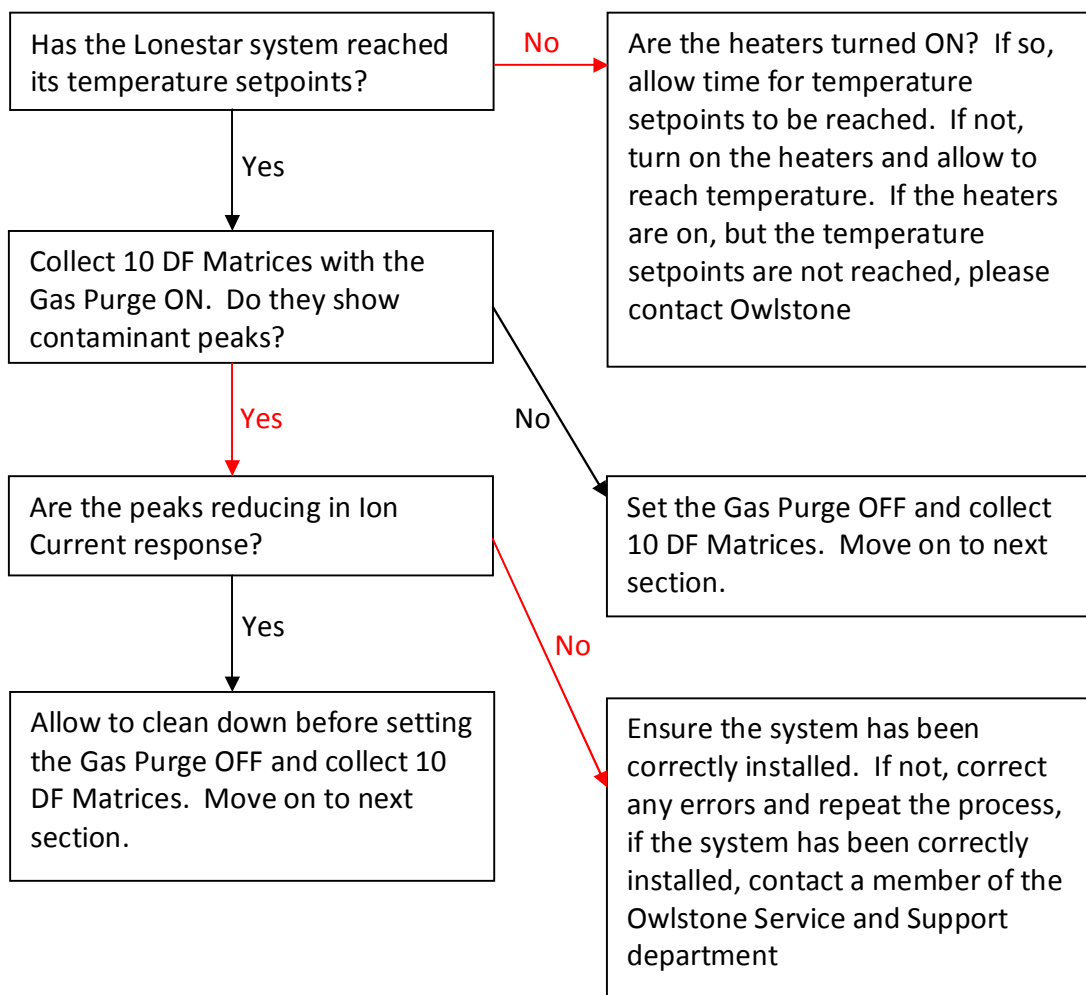
Appendix 2. Flow-chart of diagnosing contamination due to a sample being analysed is a flow-chart summarising the procedures detailed in this section of the procedure.



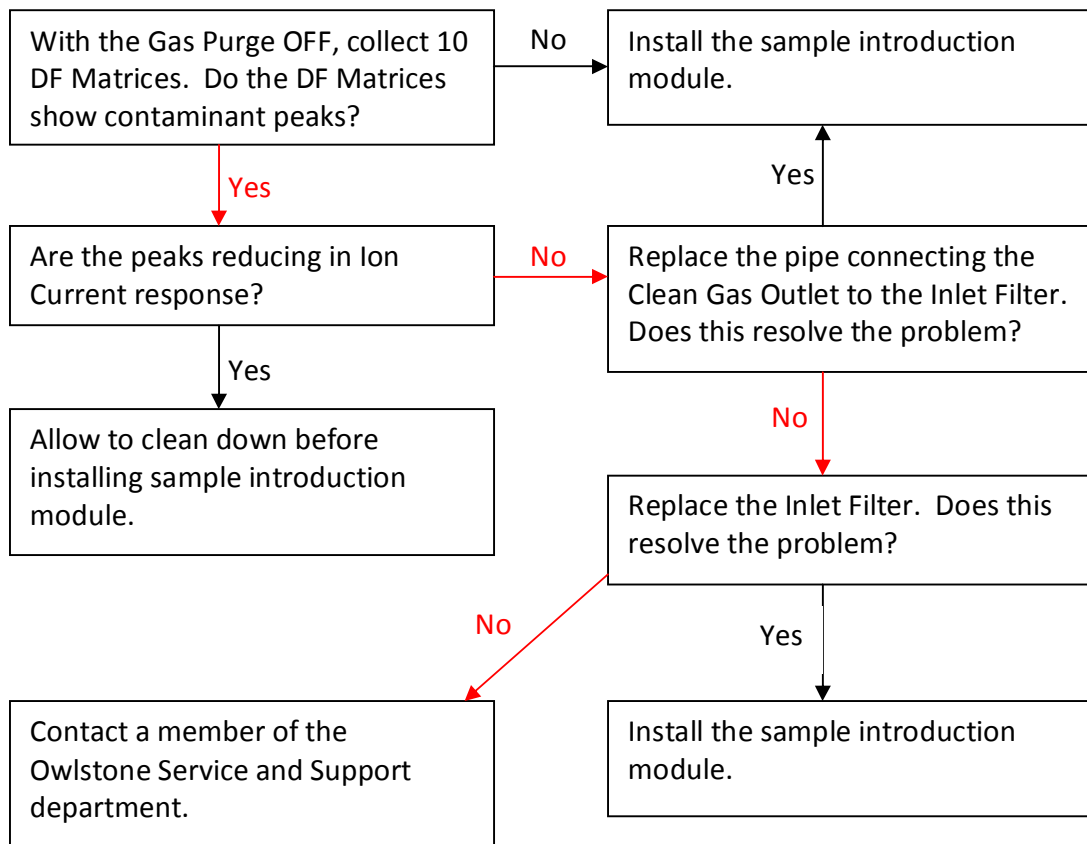
Appendix 1. Flow-chart to ensure the Lonestar system is clean on installation

Install the Lonestar system with the carrier gas passing through the pressure regulator, toggle valve and Lonestar scrubber. Install the needle valve on the Exhaust Port. Connect the Clean Gas Outlet to the Inlet Filter.

Start the Lonestar device and wait for the system to achieve its temperature setpoints.



This flow-chart ensures the internal Lonestar flowpath is clean. Once clean, set the Gas Purge OFF and collect 10 DF Matrices. The ideal path through the flow-chart is the black arrow route, with the red arrow route required to troubleshoot contamination peaks.



This flow-chart ensures the Lonestar filter flowpath is clean. Once clean, install the sample introduction module and ensure it is clean. The ideal path through the flow-chart is the black arrow route, with the red arrow route required to troubleshoot contamination peaks.

Appendix 2. Flow-chart of diagnosing contamination due to a sample being analysed

If the Lonestar system is contaminated with a high concentration of matrix/analyte peaks, this flow-chart summarises the steps to isolate the site of contamination. Always replace parts closest to the sample so as not to contaminate clean parts with the gas flow from contaminated parts.

