

Techniques for detecting and quantifying fugitive emissions – results of comparative field studies

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# Techniques for detecting and quantifying fugitive emissions – results of comparative field studies

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#### ABSTRACT

Sniffing and Optical Gas Imaging (OGI) methods for the detection of refinery fugitive VOC emissions were compared under field conditions. Both of these methods are described as Best Available Techniques (BAT) in the EU BAT Reference (BREF) document for refining of mineral oil and gas. Sniffing is a traditional method defined by standards and OGI is a newer technique offering considerable advantages. The comparison of these methods was made quantitative by using a bagging technique to explicitly measure emission rates. It was found that Optical Gas Imaging was very successful in finding the majority of the mass emissions and therefore it can be considered as effective as Sniffing and as a standalone leak detection and repair method.

#### **KEYWORDS**

LDAR campaigns, Optical Gas Imaging, OGI, Sniffing, Method 21, Leak / no-leak, refineries, field measurement campaigns

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#### SUMMARY

In 2012-2013, Concawe carried out several parallel leak detection and repair (LDAR) campaigns at two European refineries in units handling gas and light hydrocarbons. A pilot scale campaign was carried out in Site 1 and a full scale campaign in Site 2. Two techniques, namely Optical Gas Imaging (OGI) using an infra-red camera and Sniffing, were applied by different teams to detect the leaks independently. The goal was to compare the volatile organic compound (VOC) mass emissions detected by each method.

Site 1 is a newer facility, built in the 1980's, where LDAR was applied for the first time during this survey. Site 2 is an older facility with an LDAR programme in place for 10 years. A single campaign was done at Site 1 while three consecutive campaigns were done at Site 2. In the first campaign at Site 2 several units were surveyed, totalling 26,000 LDAR points. In the subsequent campaigns only sub-unit 2 was surveyed (selected as previous surveys had shown this to have a relatively high number of leaking components). Site 1 and Site 2 sub-unit 2 had approximately 4,500 LDAR points each. The leak definition threshold for the Sniffing technique was 10,000 ppmv for Site 1 and 5,000 ppmv for Site 2 (based on the site environmental permit).

In this report the word "leak" is used in three ways: (i) as a generic word for a source of emissions from a non-tight component, (ii) to describe a source of emissions where the sniffing concentration is greater than the site definition threshold and (iii) as an emission visualised using the OGI camera. The word leak should therefore be interpreted according to context.

For the detection of leaks the use of an OGI camera according to the Dutch guidelines [15] and Sniffing according to EN 15446:2008 [4] (a modified version of US EPA Method 21 (EPA-453/R-95-017) [8]) were applied. For the quantification of the leaks direct bagging methods (high flow sampling (HFS) and vacuum bagging) as well as calculation based methods, i.e. Method 21 factors and correlations and OGI leak/no-leak factors, were used.

During the field study in Site 2 a separate experiment was performed to compare vacuum bagging and high flow sampling and the deviations obtained in this comparison were further investigated in a controlled leak study. The final outcome of the controlled studies is that both bagging methods gave a relatively good agreement with the real mass leak rates.

The comparison of survey results largely confirms the known fundamental differences between OGI and Sniffing. The study established that, for the surveyed sites, either method is acceptable because the majority of the leaks were observed by both methods and both methods identified leaks that the other did not. Leaks detected by both OGI and Sniffing (the "common" leaks) represented the largest portion of the total VOC mass emissions. The results from the two sites indicate that by repairing the leaks found by OGI between 55-90% of the total VOC mass from accessible components can be abated. The higher decrease can be achieved in situations where fugitive emissions are a significant source of VOC losses. Because Sniffing and OGI are equivalent in terms of detected mass of accessible leaks, both Sniffing and OGI surveys can be applied as a stand-alone method.

A controlled leak test showed that leak rates of 0.2 g/h can be seen with the OGI camera. In real conditions OGI was able to find the majority of leaks above 1.5 g/h,

but the OGI field detection capability cannot be defined by one single value; rather the probability of the operator finding a leak increases rapidly with increasing leak size. Moreover, the effectiveness of field detection is dependent on the meteorological (e.g. wind, temperature) and other site specific conditions. In Site 1 OGI found all leaks above 21 g/h and as low as 0.1 g/h. In Site 2 all leaks above 43 g/h were detected by OGI and smaller leaks down to 1.2 g/h were detected as well. Taking into account the accuracy of the mass estimation with HFS, the performance of the OGI was equivalent (same range) for both sites.

Based on the results of this Concawe study, the OGI leak/no-leak factors [13] for 6 g/h camera leak detection sensitivity seem to be the most suitable for the overall VOC mass emissions estimation for refinery sites. This study also showed that the emissions estimated by the Method 21 factors and correlations are conservative for a facility where no leaks above 200 g/h are present. The Method 21 factors and correlations were established many years ago when the occurrence of large leaks was statistically more frequent. This method has not been revised in 20 years and could misrepresent the current situation, where LDAR programmes and technology advances (e.g. improved valve packing) have resulted in reduced fugitive emissions.

The results of the campaigns showed that the most significant leaks were identified by both methods and therefore it is not necessary to use both OGI and Sniffing in parallel. Instead, the OGI method can be used as a stand-alone method for LDAR surveys.

HFS was used to determine the mass of the leaks in order to compare the different estimation methods. However, due to the effort required for equipment bagging, bagging techniques should not be considered standard practice for regular LDAR surveys.

The size and distribution of the leaks found in the two sites were very different. Therefore the recommendations given in this report can be considered representative for European refineries.

#### 1. INTRODUCTION

The petroleum refinery industry has successfully reduced its emissions of non-methane volatile organic compounds (NMVOC), one of the precursors to surface level ozone formation, by focusing on reduced venting, vapour recovery and better storage controls. In order make further reductions, the industry is now focusing its efforts on the control of fugitive emissions (leaks)<sup>1</sup> which can contribute up to one third of the remaining site NMVOC emissions. Fugitive emissions are generated at plant components which are supposed to be leak-tight (like pump or compressor seals, valve packing, flanges, sample points, etc.). Whilst a typical site would have 50,000+ such components, only a few of these contribute to the bulk of fugitive emissions. Identifying these few leaks for repair is difficult and time consuming, as they are spread out over the entire site, including hard to access locations.

Two methodologies are currently available to detect leaking equipment in so-called LDAR (Leak Detection and Repair) programs:

- Method 21 (i.e. Sniffing), uses a hydrocarbon ionisation detector; this methodology was developed by the US-EPA and was the first historically. It is a widely accepted method, key elements of which are adopted in the European Standard EN 15446:2008 [4].
- Optical Gas Imaging, (OGI) uses an infra-red camera. It is the newer technique and gaining increasing acceptance.

Both methods are effective and each has advantages and limitations. However, as they are based on different technologies and applied in the field in a different way, comparison is not straightforward.

Although OGI is considered a new technique, OGI cameras have been on the market for more than 15 years and the technology has been improved during recent years. Because the initial detection limit of the first generation OGI cameras was higher than the sensitivity of the conventional Sniffing methods it was not considered as efficient a technology for leak detection and quantification. However the camera models currently commercially available have improved detection limit capabilities (for example by providing a "high sensitivity" operation mode) and there is much more experience with their use. A full field comparison between the techniques had not been carried out with the newest camera models. The purpose of this Concawe study was to perform such a comparison test. During 2012-2013 Concawe carried out several parallel LDAR campaigns in units handling gas and light hydrocarbon situated in two European refineries (Site 1 and Site 2). Both OGI camera and Sniffing detection methods were applied by two independent teams. The objective was to compare the number of leaks and the estimated volatile organic compound (VOC) mass emissions detected by each method. Site 1 is a newer facility, built in the 1980's, where LDAR was applied for the first time during this survey. Site 2 is an older facility with an LDAR program in place for 10 years. A single campaign was done at Site 1 while three consecutive campaigns were done at Site 2.

<sup>&</sup>lt;sup>1</sup> In this report the word leak is used in three ways: (i) as a generic word for a source of emissions from a non-tight component, (ii) to describe a source of emissions where the sniffing concentration is greater than the site definition threshold and (iii) as an emission visualised using the OGI camera. The word leak should therefore be interpreted according to context.

When possible, the mass emissions were independently estimated for all detected leaks by using a bagging technique (HFS). Additionally, for a sample of leaks another bagging method (vacuum) was applied. This vacuum method had been used for the determination of the Method 21 correlation and factors. A separate controlled experiment was also performed in order to compare vacuum bagging and HFS.

In this Concawe report, the following areas are covered:

- Description of the detection and quantification methods used
- Analysis of the field measurement results

Neither sniffing nor (at the time of this study) OGI techniques measure mass emission rate. Correlations based on multiple bagging measurements are well established for sniffing but individual component emissions are uncertain. Using a concentration threshold to classify components as Leak/No-Leak for repair purposes yields some false positives (actual emission much smaller than correlation value) and false negatives (actual emission much larger than the correlation value). OGI allows the relative size of the emission to be assessed and hence has a potential to guide a more targeted and robust repair strategy by avoiding such false positives and negatives. Unfortunately, further work to improve the quantification of OGI is needed to take this to the point where accurate repair/no repair decisions can be made on individual components. This report therefore does not address repair criteria in connection with OGI.

The report also provides recommendations on the use of OGI for leak detection and quantification.

### 2. VOLATILE ORGANIC COMPOUNDS (VOCS)

#### 2.1. VOC DEFINITION

For the purpose of this study the term VOC is considered to be defined as in the standard EN 15446:2008: "all products of which at least 20% m/m has a vapour pressure higher than 0.3 kPa at 20°C. For the petroleum industry this includes all light products and excludes kerosene and all higher (i.e. heavier) product". <sup>2</sup>

The streams concerned in these studies do not contain methane so strictly the study addresses non-methane volatile hydrocarbons (NMVOC).

#### 2.1.1. DIFFUSE VOC EMISSIONS

Diffuse VOC emission are defined by the Best Available Technique Reference Document for the Refining of Mineral Oil and Gas (REF BREF) [6] to be:

"Non-channelled VOC emissions that are not released via specific emission points such as stacks. They can result from 'area' sources (e.g. tanks) or 'point' sources (e.g. pipe flanges)"

In the descriptive section on VOC monitoring (3.28.1.4 of the BREF) examples are given:

"Diffuse VOC emissions are emissions arising from direct contact of gaseous or liquid volatile organic compounds with the environment (atmosphere, under normal operating circumstances).These can result from:

- Inherent design of the equipment (e.g. uncovered oil/water separators);
- Operating conditions (e.g. non collected vent of a fixed roof tank during loading); or fugitive emission caused by an undesired gradual loss of tightness from a piece of equipment and a resulting leak. Fugitive emissions are a subset of diffuse emission."

The focus of this report is on comparing two detection methods for fugitive emissions from point sources which typically make up between 20-50% of the overall refinery diffuse emissions. Emissions from point sources include leaks from components which are not fully sealed: pipe flanges, valve stems, pump and compressor seals, etc.) [5].

<sup>&</sup>lt;sup>2</sup>Note that the definition of VOC in the Industrial Emissions Directive (IED, 2010/75/EU) is slightly different from the EN 15446:2008 definition: "as any organic compound as well as the fraction of creosote, having at 293.15 K a vapour pressure of 0.01 kPa or more, or having a corresponding volatility under the particular conditions of use."

#### 3. LEAK DETECTION AND QUANTIFICATION METHODS

#### 3.1. LEAK DETECTION AND LEAK QUANTIFICATION

When discussing the monitoring and reporting of VOC emissions, three different purposes have to be taken into account: leak detection, identification and quantification. For the point sources considered in this report detection and identification are synonymous.

- Leak detection/identification: Different types of instruments can be used for the VOC leak detection: e.g. Optical Gas Imaging (OGI), flame ionisation detector. The number of leaks and a leak indication (e.g. measured concentration (screening) value or OGI video image) are recorded.
- <u>Leak quantification</u> is the estimate of the amount of VOCs emitted (i.e. t/a) for reporting and tracking purposes. Several methods are available: indirect (e.g. correlation equations based on measured concentrations, emission factors) or direct assessment by bagging (e.g. high flow sampling, vacuum bagging).

Leak Detection and Repair (LDAR) programmes have been put in place across European refineries in order to detect and reduce the VOC fugitive emissions. Although the main purpose of an LDAR program is to decrease VOC emission, leak quantification was added for reporting purposes and for tracking the long term progress.

#### 3.2. LEAK DETECTION METHODS

Two main methodologies are currently available to detect the emissions from leaking equipment:

- <u>Methodologies based on Sniffing:</u> the detection is done by drawing an air sample past a hydrocarbon ionisation detector to detect the VOC concentration in the vicinity of the leak source (called screening value). This methodology was first developed by the US Environmental Protection Agency (EPA) and is referred to as "Method 21". The European LDAR Standard EN 15446:2008 is a modified version of Method 21 where the frequency of the surveys and the leak repair threshold are not fixed but can be adapted based on analysis of the previous survey.
- Methodologies based on "Optical Gas Imaging": OGI uses a passive mid-wave infra-red camera for rapid and effective leak detection/identification. The camera is equipped with a filter to specifically detect hydrocarbons and the leak is visualised as an image of a plume. In 2013, the Dutch standardization institute developed national guidelines for performing detection surveys using OGI [15] and an EN standard is under development [14].

Today regulatory authorities in many EU countries have developed their own LDAR requirements, based on EN 15446:2008, OGI or a combination of these two. The REF BREF

[6] considers both Sniffing and OGI as best available techniques for the monitoring of fugitive VOC emissions to air. The REF BREF formally requires LDAR programs to take place and the use of Sniffing, OGI and calculation methods to assess emissions.

**Figure** 1 illustrates the reduction in fugitive VOC emissions achieved over several years for one particular industry case through LDAR programs [12]. In the **Figure** 1 each cycle is completed every 5 years.





#### 3.2.1. Sniffing detection instruments

Many different types of Sniffing analysers can be used to detect fugitive VOC emissions. The most common types are flame- or photo-ionization detectors (FID, PID) and infrared absorption monitors. The choice of the instrument type should be based on the type of chemical species to be surveyed. [5]

#### Flame-ionisation and photo-ionisation detectors

lonization detectors operate by ionizing the gas sample and then measuring the charge (number of ions) produced. PIDs use ultraviolet light and FIDs use a flame to ionize the organic vapours.

A FID has a well-established response to a wide range of compounds (e.g. alkanes, olefins, and aromatics) of interest in a refinery location. The response of a PID can vary significantly with double bonded compounds. Therefore the FID is most commonly used in refinery LDAR surveys. According to the US and CEN methods, FID analysers have to be calibrated for a hydrocarbon concentration range of 10 ppmv to 100,000 ppmv.

The most commonly used FID/PID type of analyser used for LDAR, the TVA-1000B, is described in Annex A. The TVA-1000B was used for the Sniffing surveys in this Concawe study in FID mode. The TVA-1000B has a dynamic range of 0 to 50,000 ppmv of methane per manufacturer specifications.

#### Infrared absorption monitor

An infrared absorption monitor measures light absorption characteristics of gases. These instruments are generally used for streams of specific chemical species at high concentration. [5]

#### Dilution probe

The concentration of hydrocarbons emitted from a leak continuously reduces as it mixes with air. A probe may therefore measure different concentrations depending on where the measurement is made and depending on the size of the release.

For several commonly used FID/PID instruments (such as the TVA-1000B), the measured concentration is linear only up to 10,000 ppmv. Because concentrations of up to 100,000 ppmv need to be measured to meet the requirements of Method 21, the range of the instrument is extended by using a dilution probe. A commonly used dilution probe provides a 10:1 dilution. [18]

The dilution probe can also be used to enrich oxygen deficient samples by adding ambient air. Low oxygen can affect the characteristics of the hydrogen flame, causing readings to be artificially elevated and possibly extinguishing the flame. More than 16% oxygen is required to support the flame.

In this Concawe study, dilution probes were used to enable measurement of higher concentrations.

#### Response factors

The detectors (FID, PID) used to obtain the screening values are calibrated with methane (FID) or isobutene (PID). However, the detector will respond differently to other hydrocarbon compounds and a correction to the calibration is required. Therefore, a response factor has to be applied to adjust an instrument reading from ppmv of methane equivalent to ppmv of total organic compound(s) before the quantification method correlations are used. Response factors are described in more details in Annex A. Use of the response factors might cause some uncertainty to the screening value if the hydrocarbon composition is unknown.

In this Concawe survey the response factors for the TVA-1000B screening values were applied at both sites. The site LDAR database was used to determine the gas or liquid composition in the equipment pieces surveyed.

#### 3.2.2. Optical gas imaging (OGI) camera

The OGI technology uses a hand-held infrared (IR) camera to detect the leaks. OGI cameras are passive mid-wave infra-red cameras equipped with a filter to selectively detect radiation at the specific C-H absorption band (3.2-3.4  $\mu$ m). Hydrocarbons that can absorb infrared radiation in this spectral range include a large number of aliphatic and aromatic compounds. OGI technology, depending on the size of the leak (mass of emissions), can be used to detect leaks from one meter to many meters away in a petroleum refinery process unit setting. The commercial OGI cameras are easy to use and show the hydrocarbon leak as a plume coming from the emitting source. Because of the remote detection capability OGI can find emission plumes from sources not otherwise accessible to the operator, so that these leaks can be detected and repaired.

In 2014 two main companies provided commercial cameras: FLIR was the first manufacturer and dominates the market. OPGAL is a new player since 2010. Based on the feedback from several contractors performing OGI surveys, the newest camera models from each manufacturer (FLIR GF 320 and OPGAL EyeC) give comparable results in the field.

The OGI camera model used in this study to visualise refinery equipment leaks was the FLIR GF-320. For more information see reference [11].

#### 3.2.2.1. OGI camera leak detection limit

The given lower detection limit for the OGI camera is 0.2-10 g/h, depending on the hydrocarbon and surroundings (e.g. temperature difference between the leak and the equipment or background).

To assess the lower detection limit of the OGI camera, Concawe performed a controlled leak test in 2013 in an European LDAR test installation. This is a facility for LDAR training and studies in which leaks of different size and from different types of equipment types can be generated. The test was performed according to the EPA-AWP. [7]. The aim of the test was to detect which flow rates (g/h) were visible using the OGI camera at 2 meters distance and

with the camera in normal or in high-sensitivity mode. Propane was used to generate the known leak rate and an open ended pipe was used as the leaking source. The sensitivity of the camera was tested from two different viewing angles: from the optimal angle towards the clear sky and from a non-optimal angle towards the dark internal wall of the test facility. The following results were obtained:

• OGI detection limit from optimal measurement angle with

∆t\*= 1.9°C (17.2-15.1): 0.2 g/h

• OGI detection limit from non-optimal measurement angle with

∆t\*= 0.3°C (15.0-14.7): 1.0 g/h

\*  $\Delta t$  was read with a given setting of the OGI camera (e.g. gain or focus) and provides a qualitative assessment of the contrast in the video.

From the optimal viewing angle the temperature difference between leak plume and the background is bigger (1.9°C) than from the non-optimal angle (0.3°C). A larger temperature difference makes the leak plume more visible and a lower detection limit can be achieved.

Results were in the same range as those reported from other controlled experiment results given in **Table 1** and in Annex B. The FLIR OGI camera model used during the Concawe field measurement campaigns was a newer model than the cameras for which the test results in **Table 1** are given. The newer model cameras have an additional "high sensitivity" mode that can be applied resulting in lower detection limits.

	Wind speed		
	0 km/h	3,2 km/h	8 km/h
Detection limit	g/h	g/h	g/h
Methane	0.8	2	6
Propane	0.4	1.3	1.3

Table 1IR-camera detection limit wind tunnel test results with different wind<br/>speeds [10]

During the field tests, a daily record was made of the meteorological conditions (e.g. wind speed, degree of sun or rain, etc.). Before the start of the OGI measurements, the sensitivity of the camera was checked. Controlled leaks were generated with propane to check that a leak of this gas of 6 g/h could be detected from 2 meter distance, this being the normal screening distance during the surveys. In addition, a detection check for a leak of 60 g/h at a distance of 10 m (this being the maximum distance for the screening of non-accessible sources) was undertaken. The same checks were also made with propylene.

#### 3.3. LEAK QUANTIFICATION/ESTIMATION METHODS

#### 3.3.1. Leak emission estimation based on the Sniffing techniques

The Sniffing technique involves placing a detecting instrument probe close to the surface of a piece of process equipment where there is the potential for a leak (e.g. at flange seal). The VOC concentration of the leak is measured by moving the probe along the surface. The maximum instrument reading in ppmv is recorded. This is referred to as the "screening value". A record is also made of the type of equipment device (valve, flange, pump seal etc.). A leak is considered to occur when the screening value measured is above a given concentration (e.g. 10,000 ppmv). The leak definition criterion can vary from one site to another and is usually set in the environmental permit. Above that given concentration threshold the equipment is identified as leaking and must be repaired. Components which give screening values below the leak definition are considered as non-leakers and repairs are not required.

This detection method requires every potential leaking point included in the database (a listing of all sources) to be surveyed and therefore this procedure is very expensive and labour-intensive.

The equipment to be monitored by Sniffing is listed in a database and is restricted to:

- Accessible points (e.g. not under insulation, able to be reached without scaffolding).
- The lines containing a light hydrocarbon (20% of the fluid m/m has a vapour pressure higher than 0.3 kPa at 20°C).

According to the EPA "Leak Detection and Repair – A Best Practices Guide" [9] the common problems and factors affecting leak detection by Sniffing are:

- Not following Method 21 properly.
- Failing to monitor at the maximum leak location.
- Not monitoring for long enough to identify a leak.
- Holding the detection probe too far away from the component interface. The reading must be taken at the interface.
- Not monitoring all potential leak interfaces.
- Using an incorrect or an expired calibration gas for the detection instrument.
- Not monitoring all regulated components.
- Not completing monitoring if the first monitoring attempt is unsuccessful due to equipment being temporarily out of service.

The other external influences affecting leak detection by Sniffing are e.g. the ambient temperature and the relative humidity. [16]

To derive the mass emissions, two commonly used quantification methods are applied:

- EPA "Screening Ranges Approach" (leak /no-leak) factors.
- EPA "Correlations Approach" (commonly referred as "Method 21").

#### 3.3.1.1. EPA Screening Ranges Approach (Sniffing leak / no-leak method)

In the Screening Ranges Approach (previously known as the leak/no-leak approach) the screening value is considered as a direct indication of the leak rate. A mass leak rate is attributed to leaks presenting screening values below 10,000 ppmv and a different leak rate is attributed to those presenting screening values above or equal to the 10,000 ppmv. The emission rate factors depend on the type of component where the leak is found. [8]

The refinery emission factors for these two ranges of screening values are presented in **Table 2.** 

Equipment type	Service	≥10,000 ppmv emissions factor (kg/h/source)	<10,000 ppmv emissions factor (kg/h/source)
	Gas	0.2626	0.0006
	Light liquid	0.0852	0.0017
Valves	Heavy liquid	0.00023	0.00023
	Light liquid	0.437	0.012
	Heavy		
Pump seals	liquid	0.3885	0.0135
Compressor seals	Gas	1.608	0.0894
Pressure relief valves	Gas	1.691	0.0477
Connectors	All	0.0375	0.00006
Open end lines	All	0.01195	0.0015

#### Table 2 Refinery screening ranges emission factors [8]

#### 3.3.1.2. EPA Correlation Approach (Method 21)

The monitoring and emissions estimating methodology 'Method 21' is described in EPA4-453/R95-017 (US). The correlation equations or factors used to estimate the emissions from leaking components originated from the 1995 US EPA Protocol for Equipment Leak Emission Estimates. In order to use the correlation equations the screening value and component type are required. The correlation equation can be applied to leaks with a screening value (SV) in the range of 1 ppmv to 100,000 ppmv. For screening values above 100,000 ppmv, the correlation is not valid and a simple factor (pegged-value) is used to determine the leak emission rate.

The correlation equation applicable to screening values between 1-100,000 ppmv:

Leak rate (kg/h) = correlation factor 1 × (SV) <sup>correlation factor 2</sup>

The equation applicable to screening values  $\geq$ 100,000 ppmv:

Leak rate (kg/h) = pegged value emission factor (kg/h)

Method 21 correlation factors and the corresponding curves for different equipment pieces are given in **Table 3** and **Figure 2**.

Table 3Method 21 correlation and pegged factors for different equipment pieces<br/>(pegged values emission factors are applied for the screening value ≥<br/>100,000 ppmv). [8]

Source	Pegged values emission factor kg/h	Correlation Factor 1	Correlation Factor 2
Open-End	0.079	2.20E-06	0.704
Valves	0.14	2.29E-06	0.746
Flange	0.084	4.61E-06	0.703
Connectors	0.03	1.53E-06	0.735
Pump seals	0.16	5.03E-05	0.61
Other	0.11	1.36E-05	0.589



# The Method 21 correlations presented in **Table 3** and **Figure 2** were developed by vacuum bagging leaks from individual pieces of equipment and comparing the emission rate with the screening value. The bagging was carried out for several equipment pieces of the same equipment type (valve, pump, connector, etc.) and for a range of service conditions (gas, light liquid or heavy liquid). The resulting correlations allow the mass leak rate to be predicted from a screening value for the equipment types and services in the database.

Figure 3.



A spread of screening value data with associated mass leak rates for

When deriving the Method 21 correlations, a large degree of variation was found: several same screening values could have represented mass emission rates with several orders of magnitude difference. The accuracy of the correlation for predicting the emission from a single source is poor. In LDAR surveys and compared to the correlation there are so called "false negatives" (large leak with low screening value) and "false positives" (tiny leak with

Base 10 Log Emission Rate (Log kg/hr)

high screening value). However, as these results apply to all leaks marked for repair, the individual uncertainty is less important and total mass emissions should have a closer agreement to the real emissions due to averaging. This is why the Method 21 correlations are only statistically meaningful if applied to a very large number of leaks. The accuracy of the Method 21 estimations for the number of leaks detected in the surveys described in this report is not as high as when full site surveys are undertaken. [8]

The leak/no-leak method described in section 3.3.2.1 is a much faster leak quantification method because only two leak categories are used. Method 21 is a more sophisticated method and more accurate results will be obtained, but it also requires the exact screening value for all the leaks to be recorded, which is more time consuming. In this Concawe study, Method 21 correlations were used when Sniffing was applied.

#### 3.3.2. Leak emission estimation using OGI techniques

The OGI surveys were performed according to the Dutch guidelines [15]. The FLIR GF 320 camera was used and the equipment was surveyed with the camera at no more than 2 meters distance from multiple angles. The pace of the survey was 2000 components per person per work day.

In the OGI surveys the camera operator scans all equipment in accordance with the instrument manufacturer's instructions. Generally, this consists of the operator viewing the equipment from different positions and in different camera operating modes. Where a leak occurs, the plume is seen as an image on the camera screen. During an LDAR survey all leak plume images or videos are recorded.

Factors affecting leak detection are ambient conditions (e.g. temperature), adequate training, amount of time viewing a component, viewing angles and distance, type of VOC (degree of absorption in the spectral range of 3.2 to 3.4  $\mu$ m) and mass emission rate. If a leak is not emitting at a sufficient rate it will not be visible using the imaging equipment. The OGI method allows the user to check the relevant sources from a distance: there is no need to check closely every potential leaking point listed in the database.

FLIR camera technology is described in more details in reference [11].

Figure 4

An image of a leak otherwise not visible to the naked eye



The US Alternative working practice (AWP) [7] considers OGI as an alternative to the current LDAR practice (Method 21 i.e. Sniffing in Europe).

#### 3.3.2.1. OGI leak / no-leak method

The American Petroleum Institute (API) developed a methodology to quantify VOC emissions rates based on the detection of the leaks by the OGI technology. The emission factors were derived for valves, pumps and flanges for a selected range of OGI camera leak detection thresholds (OGI detection limits depending on real conditions) ranging from 3 to 60 g/h. The so called OGI leak/no-leak quantification method uses a set of emission factors covering different types of equipment as shown in **Table 4**. The factors used depend on the ability of the camera to detect a controlled leak e.g. 6 g/h, which should be tested for each survey. Alternatively sites can use a single factor of 60 g/h for each leak observed. However, recent studies [16] suggest, supported by this Concawe programme, that this leads to a too high emission estimate and that a smaller leak/ no-leak factor of 6 g/h results in a better agreement with the results obtained by applying the Method 21 correlations.

The OGI "leak" factors are applied to the leaks visible as plumes on the OGI camera screen. The OGI "no-leak" factors are applied to all the other components. It is important to have an accurate count of all emission points (but a database with unique component identification is not required). In this Concawe study, OGI leak/no-leak factors were used to compare different emission estimation methods.

	Emission	Emission factor (g/h/component) for specient leak definition (g/h)			
Component type	type	3	6	30	60
Valves	no-leak	0.019	0.043	0.17	0.27
	leak	55	73	140	200
Pumps, compressors	no-leak	0.096	0.13	0.59	0.75
	leak	140	160	310	350
Flanges	no-leak	0.0026	0.0041	0.01	0.014
	leak	29	45	88	120
Other components	no-leak	0.007	0.014	0.051	0.081
	leak	56	75	150	210

## Table 4OGI Leak/no-leak method emissions factors for specified leak definitions[13]

#### 3.3.3. Method comparison– Sniffing versus Optical gas imaging

The two leak detection methods, Sniffing and OGI, have a number of differences as indicated in **Table 5**. This table was developed by the European Chemical Industry Council [3].

#### Table 5 Comparative overview of Sniffing and Optical Gas Imaging

Category	Sniffing	Gas Imaging
Detection device	Hand-held FID or PID.	Hand-held gas imaging camera.
Detection mode	Concentration measurement at every potential leak point in the field. The Sniffing probe needs to be within 1-2cm of the point where a leak can occur.	Scanning of facilities and detection of plumes. Scanning at a distance is feasible.
Applicability	All plants handling volatile hydrocarbons, particularly facilities where piping systems are easily accessible. Plants handling highly toxic substances for which very small leaks must be detected.	All plants handling volatile hydrocarbons, particularly larger facilities or facilities where many potential leak points are covered by insulation or are not easily accessible.
Result	Concentration (ppmv) in the immediate vicinity of the leak.	Video where leaks appear as plumes.
Detection limit	Depends on the nature of substances. Can detect also very low concentrations (a few ppmv) provided a suitable instrument is used.	Depends on the nature of substances. 1 to 10 g/h for aliphatic hydrocarbons and benzene.
Reliability	Occurrence of false positives (tiny leak with high ppmv) and false negatives (large leak with low ppmv).	If performed by a skilled operator <sup>3</sup> , all leaks above the detection threshold will be consistently detected.
Limitations	Accessibility: need to have close- range access to potential leak points. Not suitable for items covered by insulation. Not practical for components that are out of reach. Will detect only leaks of items included in the scope of the survey programme.	Depends on the detection limit for the substances being emitted. No limitation for accessibility. Leaks under insulation are normally detected Surveys include all potential leak sources.
Detector cost	5,000 to 20,000 €	70,000 to 100,000 €
Survey	500 components per day per	1,500 to 2,000 components per
manpower	surveyor, very labour-intensive.	day for 2-people team.
Emission quantification	Correlations between ppmv measured and kg/h leak rate; quantification of individual leaks not reliable.	Leak/no-leak factors applied to all potential leak points; quantification of individual leaks not possible.

<sup>&</sup>lt;sup>3</sup> It should be noted that a skilled operator is also required for the Sniffing surveys in order to avoid leaks to be missed by incorrect measurements

Category	Sniffing	Gas Imaging
System requirements	Emission calculation requires a database of all potential leak points.	Emission calculation requires a detailed count of the number of equipment. For speciation of emissions a database may be needed.

Based on the comparison presented in **Table 5**, the following main differences between OGI and Sniffing can be pointed out:

- Measurement result: Concentration value (ppmv) in Sniffing and visualization of the leak (i.e. video or image) in OGI method
- Accessibility: With OGI there are no limitations of accessibility. With Sniffing a certain number of components are not accessible.
- Detection limit: Lower detection limit for OGI in the field is 1-10 g/h (in the controlled release experiments 0.2 g/h level was achieved, see section 3.2.2.1) compared to approximately 0.01 g/h (on average) for Sniffing.
- Efficiency: Required manpower of OGI survey is about 4 times less than the required manpower for a similar Sniffing survey, with a strong economical advantage to using OGI.
- Cost: Cost of an OGI camera is currently approximately 5 times higher than the cost of the portable FID/PID analyser.

#### 4. DIRECT LEAK QUANTIFICATION METHODS - BAGGING

Bagging methods allow VOC leaks to be measured directly. There are currently three bagging methods:

- Vacuum bagging
- Blow through
- High flow sampling (HFS)

The correlations for the emissions quantification provided by EPA given in **Table 3** and **Figure 2** were developed based on leak quantification data collected using the vacuum bagging method.

#### 4.1. EPA – VACUUM METHOD AND BLOW-THROUGH METHOD

Bagging is defined as a means to quantify mass emissions from equipment leaks in Section 4 of the EPA Protocol for Equipment Leak Emission Estimates [8]. The Protocol defines two bagging approaches: the vacuum method (see **Figure 5** and **Figure 6**) and the blow-through method. In both methods the emission rate of a component is measured by bagging the component with an impermeable foil constructed of inert material and evacuating the undiluted leak from the bag at a constant measured flow rate. Two samples of leaking vapours are taken, using a sampling train for subsequent analysis.

The methods differ in the ways in which the carrier gas is conveyed through the bag: In the vacuum method, a vacuum pump is used to pull air through the bag. In the blow-through method, an inert carrier gas is blown into the bag. [8]

The following formula is used to calculate the vacuum bagging flow rate.

Leak Rate 
$$(g/h) = \frac{9,63 \times 10^{-10} (Q)(MW)(GC) \times 1000}{T + 273,15}$$

where

- Q = Flow rate out of Bag (L/min)
- MW = Molecular weight of molecular compound (propane= 44.1 g/mole)
- GC = Sample bag organic compound concentration (ppmv)
- P = Pressure at the dry gas meter (mmHg)
- T = Temperature at the dry gas meter (°C)



### *Figure 5* Vacuum bagging method schematic





#### 4.2. HIGH FLOW SAMPLING (HFS)

The HFS method also uses the bagging principle to measure the leak rate. A source is bagged by enclosing it in order to collect leaking vapours in a similar manner to the vacuum bagging method but a small flow of air is allowed to flow into and through the bag to the main sampling hose of the high flow sampler.

The volumetric flow rate through the bag induced by the high flow sampler's pump is calculated from the pressure differential across an orifice plate. The leak rate is then calculated as the product of the concentration and flow rate, corrected to standard conditions.

VOC concentration can be measured using the built-in HFS sensors (Catalytic Oxidation Detector (0-5% volume CH<sub>4</sub>) and a Thermal Conductivity Detector (5-100% volume CH<sub>4</sub>)) and/or an FID/PID placed at the exhaust of the instrument. In this Concawe study an FID was used. The emissions flow is calculated by multiplying the recorded HFS volumetric flow rate and the emissions concentration.

The detectors used to obtain the emission concentrations are most commonly calibrated with methane and consequently measure emissions as methane equivalent. Therefore, a response factor needs to be applied to adjust the instrument reading from ppmv of methane equivalent to ppmv of total organic compound(s). Response factors are described in more details in Annex A. Response factors are species dependent and therefore use of the response factors add uncertainty to the measured values in the case of unknown or varying VOC composition.

The FID readings are recalculated using the response factors as explained above. For the calculation of the leak rate (TOC) with the recalculated TVA reading and HFS measurement, the following formula is used:

$$\textit{Leak rate } (g/h) = \frac{\textit{Concentration} \times \textit{MW} \times \textit{Flow}}{\textit{R} \times \textit{Temp}} \times 10^{-3}$$

where

- Concentration = Actual Concentration (after recalculation) (ppmv)
- MW = Molecular Weight
  - Flow = Flow  $(m^{3}/h)$
- R = Gas Constant (0.0820578 L\*atm\*K-1\*mol-1)
- Temp = Temperature (K)



#### Figure 7 HFS sample flow in the field



#### 4.3. BAGGING METHOD COMPARISON

**Table 6** below illustrates the main similarities and differences between vacuum bagging and theHFS protocols.

 Table 6
 The main similarities and differences between vacuum bagging and HFS protocols

Vacuum bagging protocol per EPA-453/R- 95-017	HFS as used by the LDAR company during the Concawe field campaigns
Determine the composition of material in the designated equipment component, and the operating conditions of the component.	As per vacuum bagging protocol. Information was available from database.
Obtain and record a screening value with the portable monitoring instrument.	As per vacuum bagging protocol.
Special calibration procedure for the Sniffing instrument (at 5 different concentrations within the measurement range).	Not applicable for the HFS. Was done in this program for the Sniffing instrument used at the exhaust of the HFS.
Cut a bag from appropriate material that will easily fit over the equipment component.	As per vacuum bagging protocol.

Vacuum bagging protocol per EPA-453/R- 95-017	HFS as used by the LDAR company during the Concawe field campaigns
Connect the bag to the sampling train.	No samples were taken during use of the HFS. The stream composition was estimated from the site database.
If a cold trap is used, immerse the trap in an ice bath.	Not applicable.
Note the initial reading of the dry gas meter.	As per vacuum bagging protocol – the initial reading of the high flow sampler was noted.
Start the vacuum pump and a stopwatch simultaneously. Make sure a vacuum exists within the bag.	Not applicable.
Record the temperature and pressure at the dry gas meter.	Atmospheric conditions of the day recorded.
Observe the VOC concentration at the vacuum pump exhaust with the monitoring instrument. Make sure concentration stays below the lower explosive limit.	As per vacuum bagging protocol – VOC concentration at the exhaust of the high flow sampler was observed.
Record the temperature, pressure, dry gas meter reading, outlet VOC concentration and elapsed time every 2 to 5 minutes (min).	Test done during +/-1 minute after a steady state flow reading was achieved
Collect 2 gas samples from the discharge of the diaphragm sampling pump when the outlet concentration stabilizes (i.e., the system is at equilibrium).	No samples were taken during use of the high flow sampler. The stream composition was estimated from the site database.
Collect a background bag (optional). The background VOC concentration in the vicinity of the bagged component must be < 10 ppmv.	Not applicable.
Collect any liquid that accumulated in the bag as well as in the cold trap (if used) in a sealed container.	Not applicable. No bagging was done on liquid dripping leaks.
Take a final set of readings and stop the vacuum pump.	Final set of readings taken. Total test duration was +/-1 minute after a steady state flow reading was achieved. HFS removed.
Transport all samples to the laboratory, along with the data sheet.	No samples were taken during use of the high flow sampler. The stream composition was estimated from the site database.
Remove the bag.	Bag removed
Rescreen the source with the portable monitoring instrument and record.	As per vacuum bagging protocol
Purging the bag after use.	As per vacuum bagging protocol.
Use duct tape for tightening the bag.	Not applicable.

Vacuum bagging protocol per EPA-453/R- 95-017	HFS as used by the LDAR company during the Concawe field campaigns
Based on the data collected in the steps described above, mass emissions are calculated.	Similar equation used to calculate mass flow from emission points.

In this study it was decided to use the high flow sampler to bag the identified leaks. The reason for this choice was to speed up the process and be able to bag a higher number of leaks. With vacuum bagging, a maximum of 5 leaks can be bagged in a working day by a team of two. This number increases to 20 with high flow sampling. However, as HFS is not a recognized method, additional work was performed to determine its accuracy and limitations:

- In the field, 20 leaks were bagged with both HFS and vacuum bagging methods and the estimated mass emissions were compared;
- In a controlled experiment [19], a known leak rate was generated and subsequently bagged with both HFS and vacuum bagging.

The results of these tests are given in Annex C and the conclusions are summarized here:

- 1. In the controlled experiment, leak rates between 12 and 2000 g/h propane were bagged with vacuum bagging and HFS. Below 12 g/h, the bagging methods could not be evaluated due to limitation of the equipment generating the controlled leak rates; a lower accuracy can be expected for the small leak rates. However, this is acceptable as the contribution of the small leaks to the total VOC mass emission is limited
- 2. The **vacuum bagging** gave a relatively good agreement with the real mass leak rate in the controlled experiment for the entire range tested (12 to 2000 g/h).
- 3. The **high flow sampler** also gave a relatively good agreement with the real mass leak rate in the controlled experiment, but only in the range 12 to 200 g/h. Above this range, the output of the FID (TVA-1000B instrument) used in combination with the high flow sampler to determine the leak concentration was not in the linear range and the concentration value was not reliable
  - In the controlled experiment, the highest leak rates (1000 and 2000 g//h) caused the FID instrument to flame out, even when used with the dilution probe. This was never observed in the field, implying that all leaks were less than 1000 g/h
  - In the field test, 22 of the bagged leaks were measured with the dilution probe.
     Flame out did not occur so the mass emission of these leaks was between 200 and 1000 g/h, but cannot be estimated accurately
  - In this Concawe study, a fixed mass rate of 200 g/h was assigned for these 22 leaks as well as 12 other large leaks. This decision was based on the results of the field comparison between vacuum bagging and HFS (see point 4 below). This is effectively the same as adopting a pegged value for the mass flow as is assumed in Method 21.

4. During the first campaign in Site 2, 20 field leaks were bagged with both HFS and vacuum methods. In most of the cases the mass leak rate determined by HFS was greater than obtained by vacuum bagging and therefore provided a conservative determination of the mass leak rate.

On this basis, HFS was considered acceptable as the independent leak quantification method for the Concawe study.

#### 5. FIELD MEASUREMENT CAMPAIGNS

#### 5.1. FIELD STUDY DESCRIPTION

Two equipment leak detection methods, OGI and Sniffing, and their associated equipment leak estimation methodologies were studied at two European refineries. In 2012 a pilot campaign took place at Site 1 and in 2013 a larger scale campaign took place in a different refinery (Site 2). In both cases, units handling gas and light hydrocarbons were surveyed.

The aim of this study was to evaluate the differences between the Sniffing and OGI methods used for equipment leak identification and quantification.

#### Leak identification

Leak detection using the OGI technique was compared to the traditional Sniffing method. The following leak detection methodologies were used during the field campaigns:

Sniffing methodology: 15446:2008 using a TVA1000-B (FID)

OGI methodology: Dutch guidelines [15] using a FLIR GF-320 camera.

All the potential leak sources of the unit were screened in conformance with process flow diagrams (PFDs) and piping and instrumentation diagrams (P&IDs). When a leak was detected using either Sniffing or OGI, the concentration of the leak was determined by means of a portable FID and the details about the leaking source were noted.

In this study a leak is defined as:

- An OGI visual identification of a leak, or
- a screening value equal to or greater than the site leak definition threshold (Site 1 = 10,000 ppmv and Site 2 = 5,000 ppmv), or
- a combination of detection by OGI and Sniffing with a screening value equal to or greater than the site leak definition threshold.

#### Leak quantification

The mass emissions from the identified equipment leaks determined by each method were also compared.

The following direct leak quantification (bagging) methodologies were used during the field campaigns:

 High flow sampling: In this Concawe study, the HFS method was the main method used to measure emissions and provide a reference for comparing the emission estimation by the two detection methods. The field team used the manual 2-stage mode of the high flow sampler. A flame ionisation detector model TVA-1000B was used to measure the emission concentration at the exhaust of the instrument. The HFS technique is described in more details in Section 4.2 and Annex D.

- Vacuum bagging: In this Concawe study direct emission measurement was also performed on a small sample set of components at Site 2 using vacuum bagging. The blow-through bagging method was also considered for this study but after an evaluation of the sampling parameters the vacuum method was determined to be the most appropriate. Additional details of the vacuum bagging can be found in section 4.1. The method followed the procedures detailed in the Protocol with the following exceptions:
  - Background bags were not collected since the ambient background concentration was negligible relative to the high concentration emissions from the leaking components.
  - No analyses of condensed liquids were performed because no liquid phase was present in the sampling train.

The following indirect quantification methodologies were used to analyse the field campaigns results:

- Method 21
- OGI leak/no-leak factors

#### 5.1.1. SITE 1 – PILOT CAMPAIGN

In Site 1 the work was carried out in a catalytic reformer unit (with 3,931 potential leaking components) in November 2012. This study was conceived as a pilot test in order to better design the larger scale survey in Site 2 in 2013.

Site 1 is a recent facility, built in the 1980's and LDAR was applied for the first time during this survey. Two independent companies carried out the OGI and Sniffing survey in accordance with standard operating procedures.

In **Table 7** the overview of the Site 1 field campaign is presented.

Table 7. Overview of the Site 1 field campaign

Campaign	Total number of points in the scope	Number of leaks bagged with HFS	Methods
November 2012	3,931	74	OGI and Sniffing

HFS was performed on all the leaks found that were accessible. In total 74 out of 104 detected leaks were bagged.

#### 5.1.2. SITE 2

In Site 2 the study was conducted in two process units: a catalytic reformer (Unit 1) and a distillation unit (Unit 2).

Site 2 was built in 1960's and a LDAR programme had been in place from the early 2000's following regulatory requirements based on groupings of component types. The component groupings were:

- Group 1: automatic (control) valves, pressure relief valves, check valves, pumps, compressors, and associated flanges.
- Group 2: manual valves and associated flanges.
- Group 3: other equipment and components not included in Group 1 and Group 2.

The Group 3 equipment was surveyed by Sniffing when the LDAR program was first implemented. Groups 1 and 2 are measured every 5 years (or 20% of the population per year).

The field study in 2013 was conducted in three campaigns:

- 1<sup>st</sup> campaign : June 2013
- 2<sup>nd</sup> campaign: August 2013
- 3<sup>rd</sup> campaign: November 2013.

During this study an unplanned refinery-wide shut-down and start-up occurred between the second and third campaigns. Consequently the data evaluation and comparisons of the third campaign are limited in nature as the process unit shutdown/start-up had an unknown potential to affect equipment leaks. The thermal and pressure cycling of equipment or other equipment adjustments for the purpose of shutting down or starting up typically results in an increase in the number of leaking components.

The <u>first campaign</u>, included both Unit 1 and Unit 2, and Sniffing surveys were conducted on accessible Groups 1 and 2 components; The Group 3 components were not included in the Sniffing survey. OGI surveying was carried out on all Groups 1, 2 and 3 component types on both units. HFS direct measurements were conducted on most of the leaks from the OGI surveys that were accessible.

The <u>second campaign</u> was scaled down from the first one to a fraction of the Unit 2 (Groups 1, 2, and 3 components). Only OGI was performed on those components. HFS direct measurements were conducted on most accessible leaks identified by the OGI surveys. The selected subset of components was chosen from a higher leaking area identified in the first campaign in order to collect more data to compare OGI with Sniffing. The selected subset of high leakers was also chosen because in the Site 1 pilot campaign the number of leaks found and their total mass were small, and a different type of leak population was sought for Site 2 to illustrate the existing variability within sites/units.

The <u>third and final campaign</u> focused on the same subset of components as tested in the second campaign. Sniffing and OGI imaging were conducted on Groups 1, 2, and 3 component types described above. The HFS direct measurements were carried out on both Sniffing and OGI accessible leaks.

The second and third campaigns had a smaller scope due to resource constraints. With a few exceptions, no leaks were repaired between campaigns allowing a review of the evolution of the leaks already found.

In **Table 8** an overview of the Site 2 field campaigns is presented.

Campaign (2013)	Total number of points in the scope	Number of leaks bagged with HFS	Methods	Groups
		214		G1, G2 and
First - June	25,642		OGI and Sniffing	G3*
		45		G1, G2 and
Second - August	4,692		OGI	G3
		114*		G1, G2 and
Third - November	4,692		OGI and Sniffing	G3

**Table 8**Overview of the Site 2 field campaigns

\*Only OGI was applied to the Group 3 components.

\*114 leaks were bagged but only 111 of the results could be used for this study.

#### 5.2. LEAK DETECTION RESULTS

This section reviews the results from the two methods employed for leak detection. In this section, the number of leaks found and mass emission data are only used to review the detection capability of the methods and not to estimate the mass leak rates. This latter aspect is reviewed in section 5.5.

The reported mass emissions in this section are based on the results from volumetric emissions obtained by direct measurement using the HFS. These take into account the expost finding from the controlled leak study that leaks larger than 200 g/h could not have been accurately quantified with HFS due to non-linearity in the FID concentration calibration (Section 4.3 and Annex C).

As outlined in Annex C all large leaks detected in the field were attributed a constant (capped) 200 g/h emission rate. This approach is similar to the use of pegged values in EPA Method 21. For Site 1 and for the Site 2 third campaign the leaks measured by HFS were grouped as follows:


- 1. "Capped value" > 200 g/h
- 2. 43 <sup>4</sup>- 200 g/h
- 3. 1.5 43 g/h
- 4. < 1.5 g/h

This grouping was based on the results of a controlled leak test performed after the field measurement campaigns (for more information see section 4.3) and on different OGI detection capabilities (e.g. OGI lower detection capability in site 2, see section 5.3).

## 5.2.1. SITE 1

An overview of the leak detection performed is provided in **Table 9** and **Table 10**.

Table 9	Overview of the Site 1 field campaigns
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2012 Campaign Summary											
Number Found	d Leaking	Number of Equipment components	Percent of Leaking Components								
OGI	Sniffing	OGI and Sniffing	OGI	Sniffing							
34	87*	3,931	0.85%	2.18%							

\*Number of leaks above the Site 1 leak definition threshold (10,000 ppmv)

### Table 10Site 1 leaks found by Sniffing and OGI

	Number of leaks
Both methods, Sniffing above the leak definition	
threshold*	17
Both methods, Sniffing below the leak definition	
threshold*	9
Sniffing only	70
OGI only	3
Sniffing inaccessible	5
Total	104

\*Site 1 leak definition threshold is 10,000 ppmv

In Site 1 OGI technique found 34 leaks (17 of those were bagged successfully) and Sniffing technique found 87 leaks (22 of those were bagged successfully). 39 of the 87 Sniffing leaks had pegged screening values (>100,000 ppmv).

<sup>&</sup>lt;sup>4</sup> The value of 43 g/h was selected based on the results of the analysis of the bagged leaks in Site 2.

Amongst the OGI leaks, 3 were inaccessible to Sniffing and could not be bagged. No emission estimation is possible for those leaks but given that they were seen by OGI from a longer distance than the usual survey distance for accessible components (2 meters), the leak rate of these leaks is expected to be in the range > 43 g/h.

There were 25 points where HFS was not carried out because the points were either: not accessible for bagging (15), had hot surfaces (5) or for safety reasons (6). In addition there were 35 leaks which were bagged, but their emissions were too low for the measurement. Five of these were detected by both OGI and sniffing. For further analysis presented in this report, the mass leak rate of those 35 leaks was set to 0.1 g/h. In Site 1, two leaks with 0.1 g/h leak rate were measured with HFS and therefore 0.1 g/h was selected to represent the points where the emissions were too low to be measured.

In Table 11 Site 1 leaks are grouped based on the categories listed above.

Range	Total number	Number of capped leaks identified by Sniffing as large emitters (pegged)	Number of leaks found by OGI and Sniffing	Number of leaks found by Sniffing only	Total leak g/h
>200 g/h	0	0	0	0	0
43-200 g/h	2	1	1	0	48
1.5-43 g/h	31	26	14	18	270
<1.5 g/h	41	5**	7	34*	8
sum	74	32	22	52	326

Table 11Site 1, pilot, number of leaks bagged by HFS grouped by leak size

\*One OGI only but Sniffing inaccessible leak (not in the Sniffing database) is not taken into account.

\*\*Sniffing inaccessible (not in the database): Screening value obtained from the OGI survey.

In Site 1, the number of leaks found by Sniffing was higher than the number of leaks found by OGI due to the small size of the leaks and the detection limit of OGI camera. The number of leaks in size ranges and the mass distribution of leaks is shown in **Figure 8**. The scales are chosen to match a similar analysis from the third campaign at Site 2 which is presented in the next section.

**Figure 8** clearly shows the preponderance of small leaks and that Sniffing detected nearly all the leaks in the 0.1 to 6 g/h range and all those in the 6-20 g/h range whereas OGI detected 29% of the smallest leaks and 18% in the 6-20 g/h range. On a mass basis the total leak rate is very small with OGI accounting for 55% of the total bagged emissions and Sniffing 74%, where 3 sources that were "sniffed" and found to be below the site leak detection threshold have been counted as "non-leakers". Such false negatives would be missed in a standard Sniffing campaign as discussed in section 5.3.1 and shown in **Figure 9**.



*Figure 8* Site 1, Number of sources in size range





In **Figure 10** the number of leaks found by OGI per source type are given. A major part of the detected leaks came from the three equipment types: valves, flanges and connections.





## 5.2.2. SITE 2

## 5.2.2.1. LEAK DETECTION OVERVIEW

An overview of the leak detection performed across the campaigns is provided in **Table 12**, **Table 13** and **Table 14** and **Figure 11**. The table summarizes the equipment leaks detected by campaign, unit and leak detection method.

In the first campaign only the Group 1 and 2 components were surveyed by both methods; consequently there are no leaks detected by both Sniffing and OGI from Group 3 components. In the second and third campaigns all groups were included but only a subset of components surveyed in the first campaign. In the second campaign only the OGI method was performed on those components.

Because Group 3 components were not surveyed by Sniffing in Campaign 1, only Campaign 3 can be used for a valid comparison of the two leak detection methods.

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	Site 2, 2013 First Campaign Equipment Leak Identification Summary for OGI and Sniffing
T-11- 10	

	2013 1st Campaign Summary										
Numbe	er Found Le	aking	Number col	r of Equipn mponents	Perce Co	Percent of Leaking Components					
J	lune - Unit 1		Ju	ne - Unit 1		Ju	ne - Unit	1			
	OGI	Sniffing		OGI	Sniffing		OGI	Sniffing			
Group 1-2	91	120	Group 1-2	7,048	7,048	Group 1-2	1.3%	1.7%			
Group 3	57	NA	Group 3	3,833	NA	Group 3	1.5%	NA			
Total	148	NA	Total	1.4%	NA						
June - Unit	2		June - Unit 2			June - Unit	2				
	OGI	Sniffing		OGI	Sniffing		OGI	Sniffing			
Group 1-2	62	106	Group 1-2	11,413	11,413	Group 1-2	0.5%	0.9%			
Group 3	41	NA	Group 3	3,348	Group 3	1.2%	NA				
Total	103	NA	Total	14,761	NA	Total	0.7%	NA			

Table 13	Site 2, 2013 Second Campaign Equipment Leak Identification Summary for	or OGI
I able 13	Site 2, 2013 Second Campaign Equipment Leak identification Sum	mary io

	2013 2nd Campaign Summary										
Numb	er Found I	Leaking	Equipn	nent compo	Percent of Leaking Components						
Aug	ust - Unit	2 sub	A	ugust - Unit 2	2	August - Unit 2					
	OGI	Sniffing		OGI		OGI	Sniffing				
Group 1-2	60	NA	Group 1-2	3,650	NA	Group 1-2	1.6%	NA			
Group 3	44	NA	Group 3	1,042	NA	Group 3	4.2%	NA			
Total	104	NA	Total	4,692	NA	Total	2.2%	NA			

Table 14.Site 2, 2013 Third Campaign Equipment Leak Identification Summary for OGI and<br/>Sniffing

2013 3rd Ca	2013 3rd Campaign Summary										
Numbo	er Found Le	aking	Equipme	ent compon	Percent of Leaking Components						
Nove	November -Unit 2 sub			ember -Unit 2	2	November - Unit 2					
	OGI	Sniffing			OGI	Sniffing					
Group 1-2	52	62	Group 1-2	3,650	3,650	Group 1-2	1.4%	1.7%			
Group 3	49	60	Group 3	1,042	1,042	Group 3	4.7%	5.7%			
Total	101	122	Total	4,692	4,692	Total	2.2%	2.6%			

The results show that the percent of leaking components was higher for the Group 3 components in all campaigns. This could be due to the different nature of the sources or the 10 year interval between surveys compared to the five yearly interval for Group 1 and Group 2 components.

As defined above, leaks detected during the third campaign were grouped based on their size. The leak size grouping is given in **Table 15**.

Table 15Site 2, Third Campaign, number of leaks bagged by HFS grouped by leak<br/>size

Range	Total Number	Number of capped leaks*	Total leak q/h
>200 g/h	7	7	1,400
43-200 g/h	14	12	1,257
1.5-43 g/h	88	45	1,112
<1.5 g/h	2	0	2
sum	111	65	3,772

\*For the OGI only leaks the screening values were obtained from the information collected during the HFS

In **Figure 11** leaks identified by detection method are presented. Components that could not be accessed by the Sniffing monitoring contractors are not included in these counts. Figure 11 has the following categories defined:

- OGI and Sniffing: leaks that had been identified by both detection methods independently
- Sniffing-only: leaks that were found only by performing Sniffing and not found leaking when surveyed by OGI
- OGI-only: leaks that were found only by performing OGI inspections and not found leaking when inspected by Sniffing



### Figure 11. Site 2, Leaks identified by detection method

In the third campaign, the highest number of leaks was identified by both methods, and more leaks were found by Sniffing than by OGI, as could be expected due to the greater sensitivity of the TVA. The numbers for the first campaign look different, but are possibly biased because no Sniffing was performed on Group 3 components (about 1/3 of the database points).

OGI can identify leaks in the range of a few grams per hour. As expected when using two different leak detection methods, there were some cases where a leak was detected by one method but not by the other, as illustrated in Figure 11.

Direct measurements by the high flow sampler were not performed on the Sniffing-only component leaks in the first campaign and consequently sensitivity comparisons of the two detection methods is not possible. In the third campaign 26 of 39 leaks identified leaking by Sniffing-only were measured by the high flow sampler. The measured emission rates of these 26 components fell within the range of other leaks identified by OGI, suggesting that the emission rate itself is not the only factor affecting OGI leak detection.

If the number and mass of leaks detected by Sniffing and OGI are compared with all leaks in the same way as for Site 1 the distributions show some good similarities.

**Figure 12** shows that by number Sniffing accounts for most of the detected leaks in the mass leak bands less than 20 g/h and for this survey the majority of leaks were in the 6-20 g/h band. OGI detected a substantial proportion and almost as many as Sniffing in this size

band. As leaks get larger they get fewer and OGI performed overall better than Sniffing for leaks larger than 20 g/h.

**Figure 13** shows that by mass the relative contributions are clearly seen and the use of same scales puts the performance at Site 1 into perspective. Excluding the capped emissions, OGI accounts for 88% and Sniffing 86% of the accounted emissions. If capped emissions are included (at the value of 200 g/h) OGI accounts for 93% and Sniffing 81% of the total.



*Figure 12* Site 2, Number of Leak Sources in Size Range





## 5.2.2.2. CAMPAIGN TRENDS OF LEAKS DETECTED

In this section leak trends across campaigns are analysed. As previously stated, this study consisted of three different campaigns in 2013; however, an unplanned shutdown occurred between the second and third campaigns and, therefore, the leak difference between the first and second campaigns is the most relevant. The leak results for the third campaign are included in **Figure 14** for the purpose of completeness. For the first campaign, only the leak survey results from the sub-unit 2 are shown. Successive OGI campaigns showed good detection repeatability (65%) for leaks above the repair threshold.



*Figure 14* Site 2, sub-unit 2 leak trend by campaign for OGI leaks

There were 29 new leaks identified in the sub-unit 2 components surveyed during the second campaign. Eleven of the 29 were only observed in the second campaign while the balance (18) was identified in both the second and third campaigns.

The 16 new OGI leaks from Group 1 and Group 2 in the second campaign included eight found leaking by Sniffing during the first campaign. Screening values for those eight included four pegged screening values and four screening values ranging from approximately 5,300 to 17,000 ppmv of total organic carbon.

Similarly, 27 new leaks in the third campaign were not found to be leaking in prior campaigns.

As shown in **Figure 14**, 1/3 of the leaks were not consistently detected in successive campaigns. Not only the number of leaks varying between campaigns must be considered, but also how big (or small) these varying leaks are. The screening value of the new leaks detected during the second campaign ranged between 5,800 ppmv and pegged value with an average screening value of 24,673 ppmv (excluding the pegged values). This is comparable to the overall average leak screening value (29,013 ppmv). The major part of the first campaign leaks that were not seen in the second campaign and new leaks found during the second campaign are in the "OGI Partial Detection Zone" (see Section 5.3.2) based on the mass of the leaks.

### 5.2.2.3. MAJOR EQUIPMENT LEAK DETECTION

The component leaks detected are grouped by their respective major components categories in **Figure 15.** 

*Figure 15* Site 2, distribution of leaks by equipment type



The majority of the leaks detected came from valves. Valves are known to be the highest leaking group from historical Sniffing programmes. They have a tendency to leak because they have moving components.

## 5.3. DETECTION SENSITIVITY

In this section the leak detection sensitivity of OGI, as conducted with the GF-320 camera, and of Sniffing, as performed with the TVA-1000B are evaluated.

## 5.3.1. SITE 1

**Figure 16** plots on a log/log graph the results obtained by OGI and those obtained by Sniffing. The x-axis is the concentration determined by Sniffing and the scale is 5,000 to the pegged concentration of 100,000 ppmv. The leak definition threshold for this site is 10,000 ppmv. The y-axis indicates mass flow rates determined by bagging (with HFS). The minimum mass flow detection was 0.13 g/h.

The red triangles represent the pairings (concentration, mass flow) obtained from the leaks only found by Sniffing ("Sniffing-only leaks"). No relationship is visible between high screening value and high mass flow rate, as would be expected from the Method 21 correlations. Many leaks, with screening values in the range 10,000 - 100,000 ppmv were found to be small (around 0.1 g/h); similarly, at the pegged value of 100,000 ppmv, leak rates varied by over two orders of magnitude. The leaks detected by both OGI and Sniffing are shown as blue diamonds. The bagging of some leaks (35 in total, 4 common and 31 Sniffing only) were below the limit of measurement for the HFS, and these are shown as 0.1 g/h in **Figure 16**.



*Figure 16* Site 1 OGI detection sensitivity

Based on Figure 16, and the representation of the data in Figure 8 the following conclusions can be made if the data are grouped according to Figure 12 and Figure 13.

Site 1		Number			Mass g/h			]	Nu	mber	r	nass
Range g/h	Found by Sniffing	All leaks found	Found by OGI		Found by Sniffing	All leaks found	Found by OGI		OGI/All	Sniff/All	OGI/All	Sniff/All
0.1-6	55	59	17		69	73	41		29%	93%	56%	94%
6-20	11	11	2		110	110	16		18%	100%	14%	100%
20-43	2	3	2		62	95	74		67%	67%	78%	66%
43-100	0	1	1		0	48	48		100%	0%	100%	0%
100-200	0	0	0		0	0	0		-	-	-	-
>200	0	0	0		0	0	0		-	-	-	-
Total	68	74	22		241	326	179	]	30%	92%	55%	74%

Table 16Site 1, average mass emissions

- 1. The majority of leaks detected by OGI and Sniffing had pegged concentrations (>100,000 ppmv) but none of them was a large leak (> 200 g/h).
- 2. OGI detected all leaks above 21 g/h.
- 3. OGI detected 30% of the leaks above 0.1 g/h by number and 55% by mass.
- 4. The 3 leaks above 21 g/h were detected by both Sniffing and OGI however the Sniffing results would not have flagged two of these for repair because their concentration was below the repair threshold.
- 5. 5 leaks were found by both methods with a screening value below Site 1 leak definition (10,000 ppmv) but three of them had a negligible mass flow rate.

The weather conditions during the OGI surveys were favourable: it was sunny, 16 °C and not windy.

## 5.3.2. SITE 2

The data presented in **Figure 17** indicate the concentrations and mass flow ranges likely to be seen by OGI.

**Figure 17** shows the mass emissions and leak concentrations for leaks either identified by both OGI and Sniffing or individually by the one or the other method. The third campaign is solely used for this analysis, as it was the only campaign where both OGI and Sniffing leaks were directly measured with the high flow sampler.

The data points presented on the x-axis start at the leak definition of 5,000 ppmv and extend to pegged Method 21 screening values (i.e. >100,000 ppmv) at the far right of the chart. The y-axis starts at 1 g/h, (1.2 g/h was the lowest leak mass rate detected at Site 2).



## *Figure 17* Site 2, sub unit 2, OGI field detection limit

The smallest mass emission measured with the high flow sampler in the third Campaign was 1.2 g/h.

**Table 17** provides the average mass emission results used in Figure 17 and the data are grouped incommon format with that used for Figure 12.

Site 2 Third campaign	Number			Mass g/h			Nur	nber	Mass	
Range g/h	Sniffing	All	OGI	Sniffing	All	OGI	OGI/All	Sniff/All	OGI/All	Sniff/All
1.5-6	22	27	15	74	99	48	56%	81%	48%	75%
6-20	43	50	38	501	595	446	76%	86%	75%	84%
20-43	13	14	12	421	464	390	86%	93%	84%	91%
43-100	7	8	8	441	497	498	100%	88%	100%	89%
100-200	4	5	5	616	717	717	100%	80%	100%	86%
>200	5	7	7	1000	1400	1400	100%	71%	100%	71%
Total	94	111	85	3053	3772	3499	77%	85%	93%	81%

Table	17
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Site 2, average mass emissions

Based on **Figure 17**, the following conclusions can be made:

- The OGI All Leaks Detection Zone represents emission rates above 43 g/h. All leaks in this zone were detected by the IR camera.
- The Partial OGI Detection Zone represents emissions between 1.2 and 43 g/h. Leaks in this zone represent 73% of the total leaks and were identified either by OGI or by Sniffing. In the Partial OGI Detection Zone, leaks detected by OGI represent 76% of the total mass emissions. In the partial OGI leak detection zone 90 leaks in total were bagged; of these 24 leaks were missed by OGI and 11 leaks were missed by Sniffing. The "Sniffing-Only" leaks are distributed towards the left side where the emission concentration and mass emissions are lower. OGI did detect one leak in the left side area that had not been detected by Sniffing. While the concentration of that emission point was low, (6,000 ppmv) the mass emissions were still greater than 10 g/h. When the Sniffing survey was performed the concentration was most probably below the leak definition and due to that this point was not counted as a leak. It should be noted that the screening value can vary significantly between two individual measurement times. It should also be noted that when using the Method 21 correlations for the leak quantification this point is a false negative with low screening value and high mass emission rate.
- The No OGI Leak Detection Zone represents emission rates below 1.2 g/h, the lowest OGI-identified leak detected in this study. However, in the same meteorological conditions there were no leaks less than 1.0 g/h detected by the Sniffing method either.
- Leaks detected only by OGI ("OGI-only" leaks): Most of the 17 "OGI-only" leaks are pegged values and therefore could also have been found by Sniffing. There are a number of possible reasons for this, e.g. the Sniffing screening value was less than the leak definition (at the time of the Sniffing survey) or the components were not in the database or they were out of the scope of the Sniffing survey (e.g. outside unit battery limits) or they were not in operation.

**Table 18** breaks down the leak detection information for the two methods based on the screening values from Sniffing. The screening values for OGI-only leaks were obtained by Sniffing performed during the direct measurement with HFS. This allows the dataset to be expanded for evaluation.

## Table 18 Site 2, Screening Ranges of Equipment Identification for Third Campaign

	3rd Cam	paign: OGI vs. S	niffing		
	Pegged Screening Value	Non-Pegged Screening Value	Non- Sniffing Scr	pegged eening (p	pmv)
	Number of Leaks	Number of Leaks	Average	Min	Мах
Both Identified	54	29	45,352	5,700	96,000
OGI-only *	15	3	52,000	6,000	90,000
Sniffing- only	15	24	20,208	5,130	90,000

\*The screening values were obtained from the information collected from the HFS.

The majority of leaks detected by either of the two methods (Sniffing or OGI) were pegged readings. The minimum screening values of the non-pegged leaks are quite similar for the 3 leak categories (5,000-6,000 ppmv). The average of the non-pegged screening values is highest for the OGI-only leaks (52,000 ppmv) and lowest for the Sniffing-only leaks (20,200 ppmv). This is consistent with the OGI detection focussing on the larger leaks. This is confirmed in **Table 19**.

**Table 19** presents emissions measured by HFS for the OGI leaks and the Sniffing leaks. The results are split into pegged and non-pegged emissions. It was found that in both categories the leaks not found by OGI i.e. Sniffing-only were much smaller than the average. **Table 20** shows average mass leak rates for the third campaign bagged leaks.

Table 19	Site 2, Emission rates for OGI & Method 21 directly measured (HFS)
	leaks for third campaign

3rd Campaign: OGI vs. Sniffing - bagged leaks								
	Pegged	Pegged Emission (g/h)				gged Emis	sion (	g/h)
	Count	Average	Min	Max	Count	Average	Min	Мах
Identified by both OGI and Sniffing	43	61.1	2.1	200	28	15.4	1.7	77
OGI-only*	14	50.1	4.4	200	3	6.1	1.1	11
Sniffing-only	7	21.1	7.6	43	19	6.6	1.0	16.9

\*The screening values were obtained from the information collected from the HFS

Table 20Site 2, Average emission rates for directly measured leaks for third<br/>campaign

	Both detected	OGI-only	Sniffing-only
Average emissions rate g/h	43.0	42.3	10.5

Ambient temperatures were recorded throughout the campaigns. The difference between the first two campaigns was 2°C and between the second and third campaigns was 19°C. The changes in temperature between the campaigns did not appear to influence the ability of an OGI operator to identify leaks, as judged by the number of leaks found.

## 5.3.3. LEAK DETECTION SUMMARY

In this Concawe study both leak detection methods appear comparable. The majority of the leaks were detected by both methods and each method identified leaks that the other did not. The leaks found by both methods represent the largest portion of the total VOC mass emissions.

For Site 2, the screening range observations and the mass emissions results from the HFS both show that leaks found by Sniffing and not by OGI represent a much smaller portion of the mass than those found by OGI alone, and also by leaks detected by both methods.

The mass rate of the smallest leaks found were comparable for both OGI and Sniffing (1.2 g/h in Site 2 and 0.13 g/h in Site 1). Emissions above 1.2 g/h were regularly identified by OGI in the study.

In the cases where Sniffing did not detect leaks found by OGI, the emissions were at high concentrations and could have been detected. However, a number of these leaks were either inaccessible, out of scope, out of service or not in the monitored group. OGI has the clear advantage that it is able to detect such leaks.

The OGI detection capability in the field cannot be defined by one single value. Factors affecting the OGI detection capability are e.g. weather conditions, background temperature, operator training, viewing angle and distance to the source, type of VOC, survey speed. One explanation for the OGI finding smaller leaks in Site 1 compared to Site 2 might be the lower average wind speed at Site 1.

## 5.4. EQUIPMENT SURVEY RATE

After the completion of the leak detection surveys, the hours required to complete the OGI monitoring and Sniffing were analysed to determine the leak detection pace.

On average, in an eight-hour day, approximately 536 components were monitored by Sniffing per 2 person team.

In the same eight-hour day the OGI 2 person team screened approximately 1,850 components. The camera was also able to detect leaks from components that were not originally in the scope.

## 5.5. LEAK QUANTIFICATION

This section compares Method 21 correlations, the OGI leak/no-leak method and HFS for emission estimation. These methods are briefly explained in section 3 "Leak detection and quantification methodologies"

### 5.5.1. SITE 1

For Site 1, there were 74 component leaks that were measured by the high flow sampler and which had a calculated Method 21 (M21) value. Average and total leak rates for these are presented in **Table 21**.

		HFS	M21	HFS	M21
	Number of leaks	Total Emi	ssions (kg/h)	Average	Emissions (kg/h)
Pegged	33	0.25	3.59	0.01	0.112
Non- Pegged	41	0.08	0.132	0.002	0.003
Total	74	0.33	3.73	NA	NA

**Table 21.**Leak rate estimation for bagged leaks

For the pegged readings, the total mass emissions estimated using Method 21 correlations are approximately 15 times greater than the emissions measured directly by HFS. For the non-pegged readings the Method 21 correlations are in closer agreement. The total mass emissions estimated using Method 21 correlations are more than 10 times higher than the emissions measured directly by HFS.

**Figure 18** shows for Site 1 a comparison of the VOC mass emission estimation based on the different methodologies for the 17 bagged leaks found by both OGI and Sniffing . The methodologies compared are the Method 21 correlations, the OGI leak/no-leak factors [13] for 3, 6, 30 and 60 g/h camera specified leak definition and HFS.

# *Figure 18* Total mass leak rates estimated by different emission estimation methods for bagged leaks found by both methods



As noted above, the number of large leaks in Site 1 was very low. For this facility, with relatively low fugitive emissions, both Method 21 correlations and OGI factors gave an overestimate of VOC mass emissions.

## 5.5.2. SITE 2

For the third campaign, there were 97 leaks measured by the high flow sampler and which had a Method 21 screening value. Average and total leak rates for these data points are presented in **Table 22**.

Table 22Comparison of emissions measured by HFS to Method 21 (M21)<br/>correlation estimates for bagged leaks with Sniffing screening value in the<br/>third campaign

		HFS	M21	HFS	M21
	Number of leaks	Total	Emissions (kg/h)	Average Emi	issions (kg/h)
Pegged	50	2.77	5.17	0.055	0.103
Non-Pegged	47	0.56	0.20	0.012	0.004
Total	97	3.33	5.37	NA	NA

For the pegged readings, the total mass emissions estimated using Method 21 correlations are approximately twice that of the emissions measured directly by HFS. For the non-pegged readings, Method 21 correlations give approximately half the mass emission value measured by HFS. The non-pegged emissions are an order of magnitude smaller than the pegged emissions. Overall the Method 21 correlations over-estimated the total mass emissions rate by a factor of 2. This is a much smaller difference than for Site 1, which had a factor of 10.

**Figure 19** shows for Site 2 a comparison of the VOC mass emission (from bagged leaks only) based on the different methodologies for the leaks detected by both OGI and Sniffing (71 leaks for third campaign). The methodologies compared are Method 21 correlations, OGI leak/no-leak factors for 3, 6, 30 and 60 g/h camera specified leak definition and the high flow sampler.





The leak/no-leak factors over-estimated the VOC emissions, as did the Method 21 correlations. OGI leak/no-leak factors for 3 g/h and 6 g/h limit seemed to give a relatively reasonable VOC mass emissions estimate for Site 2 and bracket the average value determined by Method 21.

## 5.5.3. LEAK QUANTIFICATION SUMMARY

For Site 1, the total mass emissions measured by HFS were an order of magnitude lower than the mass emissions calculated by other estimation methods. As seen from **Table 10**, in Site 1 there were no large (over 200 g/h) leaks.

In Site 2, during the third campaign 8 large (over 200 g/h) leaks were measured. These 8 leaks out of a total of 114 emit 40% of the total mass measured by bagging.

For both Sites 1 and 2 the Method 21 correlations over-estimated the mass emissions. The Method 21 factors and correlations were established many years ago when the occurrence of large leaks was statistically more frequent. This method has not been revised in 20 years and could misrepresent the current situation, where LDAR programmes and technology advances (e.g. improved valve packing) have resulted in reduced fugitive emissions.

The OGI leak/no-leak factors for 6 g/h camera specified leak definition gave a reasonable and slightly conservative VOC mass emissions estimate for Site 2 and over-estimated the emissions for Site 1. It should be noted that there will be some leaks missed with either leak detection method (Method 21 or OGI) during the surveys. Therefore, a slight over-estimation using the emission factors may be considered to provide a more reasonable estimate of the total mass emissions from a site.

Two very different cases (Site 1 and Site 2 sub-unit 2) were surveyed during this Concawe study, illustrating the range of variability in fugitive emissions leaks which can be found in EU refineries, not only between sites but also between units within the same site.

The following other differences between the Site 1 and Site 2 sub-unit 2 were observed:

- In Site 1 the number of leaks was smaller than at Site 2. The leaking fraction detected by OGI was 0.85% and by Sniffing 2.18%. In addition, the leaks were smaller on average than at Site 2, with the result that OGI found fewer leaks than Sniffing. In Site 1 the repair threshold is 10,000 ppmv, but no big leaks were detected during the field campaign.
- In Site 2 (sub-unit 2, campaign 3) the number of leaks was greater than in Site 1. The leaking fraction detected by OGI was 2.2% and by Sniffing 2.6%. Leaks were bigger on average than at Site 1 and the number of leaks detected by OGI leaks was closer to the number detected by Sniffing. In Site 2, the repair threshold is 5,000 ppmv, but there were still more large leaks (>200 g/h) than in Site 1.
- The probability of leak detection by OGI increases with leak size and could be influenced by the specific conditions during the survey. At Site 1 smallest leak detected by OGI was 0.13 g/h and at Site 2 it was 1.2 g/h.
- The leaks smaller than 6 g/h did not significantly contribute to the total VOC mass. As seen from **Table 11** and **Table 15**, the total mass emissions from the smallest leak group at Site 1 was 41 g/h (23% of the total) and at Site 2 was 48 g/h (1.4% of the total).

The leak/no-leak factors for 6 g/h over-estimated the VOC emissions for Site 1, as did Method 21. They gave a reasonable and conservative estimate for Site 2, sub-unit 2. Based on the observed average field detection limit for the new camera model FLIR GF 320, when applied according to the Dutch protocol (regarding distance and survey speed), the leak/no-leak factors for 6 g/h (leak definitions, **Table 4**) seem to be an appropriate choice with which to derive an overall VOC mass emission estimation for European refinery situations

# 6. OVERALL CONCLUSIONS

This Concawe study has made an objective comparison of two different methods of fugitive emission detection. It was found that the OGI technique is efficient and effective in identifying leaks from components including those normally inaccessible to Sniffing. OGI can screen multiple components at a given time and find leaks above 1 g/h on a consistent basis.

To verify the HFS technique a controlled bagging experiment was carried out after the surveys. In the controlled leak study both bagging methods gave a relatively good agreement with the real mass leak rates.

The main five conclusions are:

# 1. Leaks that represent the majority of the total fugitive VOC mass emissions are detected independently by both OGI and Sniffing.

When applied independently in a field survey Sniffing and OGI do not find exactly the same leaks, which can be expected due to the inherent differences between the two techniques. However, the majority of the accessible leaks are detected by both techniques and these leaks have the highest mass contribution.

In Site 1, the mass of leaks detected by OGI was 55% of the total mass of accessible leaks and for Site 2 sub-unit 2, the mass of OGI leaks was 90% of the total mass. This range is in line with an analysis done in 1997 by the American Petroleum Institute [1].

# 2. Sniffing and OGI are equivalent in terms of detected mass of accessible leaks and therefore the same frequency of Sniffing surveys can be applied for OGI surveys

As stated above, Sniffing and OGI do not find the same leaks, but the majority of the mass of the leaks is found by both methods although neither method finds all the leaks. Large leaks contribute the most to the total mass of VOC emissions, while small leaks (below 1.5 g/h) have a minor impact. At the two sites surveyed, by repairing the accessible leaks detected by OGI between 55 to 90% of the total VOC mass could be abated. In situations, where fugitive emissions are a significant source of VOC, the number will be closer to 90% because of the number and magnitude of the large leaks. OGI has the additional advantage of being able to detect any other significant leaks in the area e.g. either not accessible or not listed in the equipment component database.

## 3. In real conditions, the OGI detection limit cannot be defined by one single value.

Controlled release experiments showed that the OGI camera used in this study could detect leaks as small as 0.2 g/h in good and 1 g/h in less favourable conditions. The effectiveness of field detection is dependent on the site conditions during the survey (e.g. temperature, wind, background scene), amount of time viewing a component, viewing angles and distance, type of VOC (degree of absorption in the spectral range of 3.2 to 3.4  $\mu$ m).

In Site 1 OGI detected all the leaks above 21 g/h and 77% of the leaks above 6g/h. In Site 2 OGI found all the leaks above 43 g/h and 82% of the leaks above 6g/h.

The camera model used during the field study (FLIR GF320) has enhanced optics and improved detection capabilities, for example the "High Sensitivity" mode. It was found that when the camera was used at the pace of 1,500-2,000 components per day, and the scanning was done at about 2 meter distance and from different angles for the accessible components, the majority of the smaller leaks above 1.2 g/h were detected.

# 4. Quantification – the OGI leak/no-leak factors for 6 g/h seem to be the most suitable for the estimation of the overall VOC mass emissions

The OGI leak/no-leak factors for 6 g/h [13] over-estimated the VOC emissions for Site 1 and a reasonable and conservative estimate for Site 2. Based on the observed average field detection limit for the new camera model FLIR GF 320, when applied according to the Dutch protocol (regarding distance and survey speed), the leak/no-leak factors for 6 g/h seem to be an appropriate choice for the estimation of the overall VOC mass emissions .

The 6 g/h leak/no-leak factors are therefore considered to be appropriate for use on refinery sites. This is based on the very different fugitive emissions situations observed for Site 1 and Site 2 during the field study, which illustrate the spread of conditions existing in the refining sector.

# 5. Quantification - The emissions estimated by the Method 21 factors and correlations are conservative for a facility where no leaks above 200 g/h are present.

For Site 2 sub-unit 2, the emissions estimated with Method 21 are close to those measured with HFS, being a factor of 2 higher. For Site 1, the emissions estimated with Method 21 are much higher than the HFS measurement (a factor of 10 higher). The distinguishing feature of Site 1 was that all of the leaks were small. A possible explanation is that the Method 21 factors and correlations established in 1993, on mostly uncontrolled facilities, over-estimate emissions on modern plants where, for example, high integrity equipment may be installed.

## 7. RECOMMENDATIONS

This program of four large field trials to compare the leak detection performance of OGI and Sniffing, supported by quantitative measurement of the mass rate of the detected emissions, has returned some very practical information from which the following recommendations can be made. These recommendations should be applicable across the refinery population; the units studied were very different and the techniques were applied independently and as part of real LDAR surveys.

- Sniffing and OGI LDAR surveys give an equivalent result and therefore OGI can be applied as a standalone method.
- Use of emission factors (leak/no-leak) in conjunction with OGI surveys is an effective mean of estimating site emissions. It is not necessary to carry out separate bagging exercises. OGI leak/no-leak factors for 6 g/h are recommended for the VOC emission quantification in OGI based LDAR campaigns
- Further work should be carried out to refine the application of OGI in order to achieve better discrimination of leak sizes on an individual component level and to reduce the uncertainty inherent in site-specific leak/no-leak criteria leading to better control of emissions.

# 8. GLOSSARY

API	American Petroleum Institute
AWP	Alternative Work Practice
BREF	Best Available Techniques Reference Document
EPA	Environmental Protection Agency
FID	Flame Ionisation Detector
HFS	High Flow Sampling (or Sampler)
IR	Infrared
LDAR	Leak Detection and Repair
NMVOC	Non-Methane Volatile Organic Compound
OGI	Optical Gas Imaging
P&ID	Piping and Instrumentation Diagram
PFD	Process Flow Diagram
PID	Photo-Ionisation Detector
SOCMI	Synthetic Organic Chemical Manufacturing Industries
SV	Screening value
ТОС	Total Organic Compounds
VB	Vacuum Bagging

VOC Volatile Organic Compounds

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## ANNEXES

## ANNEX A – TVA-1000B AND RESPONSE FACTORS

## 1. FID MEASUREMENTS DURING THE FIELD MEASUREMENT CAMPAIGNS

During the Concawe field measurement campaigns the emissions monitoring procedure using the TVA-1000B<sup>5</sup> comprised the following steps:

- Calibration of the TVA (according to US EPA guidelines)<sup>6</sup>.
- Performing measurements:
  - During the OGI campaigns (FID screening after OGI leak detection) or
  - During the Sniffing campaigns: measurements were performed source by source, based on the process and instrumentation diagrams (P&IDs) combined with the inventory data.

### TVA-1000B

The detection device used to perform the measurements was the TVA-1000B, which is a Flame lonization Detector (FID). An FID measures organic compounds by utilizing a flame produced by the combustion of hydrogen and air in the measurement chamber. When hydrocarbons in a sample are introduced to the detection zone, ions are produced by the following simplified reaction:

 $RH + O \rightarrow RHO^{+} + e^{-} \rightarrow H_2O + CO_2$ 

Where R = carbon compound.

A collector electrode with a polarizing voltage is also located within the detection chamber and the ions produced by this reaction are attracted to it. As the ions migrate towards the collector a current is produced which is directly proportional to the concentration of hydrocarbons introduced in the flame. This current is then amplified and sent to a microprocessor and/or analogue readout device. The unit of measurement of the TVA-1000B is ppmv; the ppmv-value is shown on the display.

The enclosure of the analyser is made from a chemically resistant thermoplastic material. The dimensions are approximately  $13.5 \times 10.3 \times 3.2$  inches. The instrument weighs 4.8 kg. It comes in a kit containing tools to access the battery and other parts. An electrode is located within the detection chamber. The dynamic range of the device goes from 0 to 50,000 ppmv of methane.

 <sup>&</sup>lt;sup>5</sup> Thermo Environmental Instruments INC.,TVA-1000B, Toxic vapor analyzer Instruction Manual P/N BK3500, 2001
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<sup>°</sup> EPA (1995) Protocol for equipment leak emission estimates. Publication EPA-453/R-95-017. Research Triangle Park: US Environmental Protection Agency

### Calibration of the TVA-1000B

To ensure qualitative readouts of the ppmv values on the detection devices, all detection devices were calibrated three times a day according to US EPA's calibration procedures. For the TVA-1000B detection device, two concentrations of methane calibration gas were used, 500 ppmv and 10,000 ppmv. No measurements were performed using a detection device that was not calibrated correctly.

In the morning, the device was calibrated. The first part of this calibration was a "zero-calibration." The detection device was calibrated to 0 ppmv, using zero air i.e. containing no VOCs. The second step was calibrating the device at 500 ppmv using a bottle of methane/air mixture. This calibration was performed under the same external conditions as the zero-calibration. Then, there was a calibration with a bottle of methane at 10,000 ppmv.

At noon, the detection device was checked if its readings were still accurate. The response factor of the device was set to default and checked with bottles of methane at 500 and 10,000 ppmv. A deviation of 5 to 10% was allowed. If the deviation exceeded 10%, the detection device had to be calibrated again. All sources above leak definition had to be re-measured after such re-calibration.

At the end of the working day, the deviation of the device had to be checked to see if it was within range. This procedure was the same as the one used at noon.

If the device showed a deviation at the first calibration, the cycle of calibration was repeated until the device was set properly. Should there still be a malfunction, a few steps could be taken to check for errors: another bottle with calibration gas could be used, certain filters could be replaced and / or the batteries of the device could be replaced. If this fails, the technician would contact the offices to report the malfunction.

The calibration gases were delivered by a third party. The third party delivered certificates to ensure the quality of the concentrations.

#### Data logging and recording

After calibration, data were recorded by the technicians, using the device described above. All data obtained were written on a hardcopy inventory. The readout leak values of all sources were added on the hardcopy inventory and monitoring documents for the information that was collected. In this way each unique Leak number was linked with the corresponding ppmv value.

## **TVA-1000B** specifications

Specifications	TVA 1000B - FID
Accuracy	$\pm 25\%$ of reading or $\pm 2.5$ ppmv, whichever is greater, from 1.0 to 10,000 ppmv.
	Accuracy is achieved using methane with a 1 point calibration in the range from 100 to 500 ppmv (including drift) at the temperature and humidity of the calibration.
Repeatability	±2% at 100 ppmv of methane
Analog Output	Two analogue output signals, 0 to 2V dc, proportional to the count output from each detector.
Dynamic Range	1.0 to 50,000 ppmv of methane
Linear Range	1.0 to 10,000 ppmv of methane
Minimum Detectable Level	The minimum detectable level is defined as two times the peak-to peak noise = 300 ppb of hexane
Response Time using close area sampler	Less than 3.5 seconds for 90% of final value, using 10,000 ppmv of methane
Recovery Time using close area sampler	Less than 5.0 seconds to return to 10% of base line, using 10,000 ppmv of methane
Response Time using telescoping wand extender	Less than 5.0 seconds for 90% of final value, using 10000 ppmv of methane
Recovery Time using telescoping wand extender	Less than 5.0 seconds to return to 10% of base line, using 10,000 ppmv of methane
Response Time using charcoal filter adapter	Less than 20 seconds for 90% of final value, using 10,000 ppmv of methane
Recovery Time using charcoal filter adapter	Less than 20 seconds to return to 10% of original value, using 10,000 ppmv of methane
Data Storage Interval	Auto Mode — 1 per second to 1 per 999 minutes VOC or FE Mode — 2 to 30 seconds, user-selectable
Sample Flow Rate	1 litre/minute, nominal, at sample probe inlet
FID Life	Greater than 2,000 hours
Gas Cylinder Capacity	Pressure — 15.3 MPa at 25°C (2200 psi at 77°F) maximum
Hydrogen Supply Operating Time	Greater than 8 hours of continuous operation, starting from a cylinder charged up to 15.3 MPa (2,200 psi)

### **Response factors**

The TVA-1000B is calibrated with methane. However, the detector responds to many different compounds with differing levels of sensitivity. In order to adjust the analyser reading from "ppmv of methane" to "ppmv of the compound of interest", a correction factor must be applied to the reading. This correction factor is also known as a "Response Factor."

As a stream of a plant can contain a wide variety of products, each with their specific physical and chemical properties, a different response factor has to be implemented in the VOC analyser to acquire the correct ppmv readout value. The different VOC analysers each have their specific characteristics but differ in operational principles.

When measurements are being performed using a TVA-1000B, each product is assigned a set of two response value factors, A and B. As an example, response factors for benzene and n-hexane are given in **Table 1**.

Product	А	В
benzene	0.35	0.23
n-hexane	0.43	0.30

Table 1Example response factors TVA-1000B

The response factors are provided for a list of pure products by the manufacturer of the detection device<sup>7</sup>. If a single compound is present, response factors can be applied to correct for the response of that compound. If a mixture of compounds is present, the TVA-1000B will respond to all components of the mixture and will not differentiate between them. In general, response factors are not available for mixtures of compounds. However, if the composition of the mixture is known, a response factor can be calculated by adding weighted fractions of the response factors of the individual compounds.

In detail, the correction factor for a mixture (weighted response factor) is calculated from the sum of the mole fractions  $X_i$  of each component of the mixture multiplied by their respective correction factors  $CF_i$ :

 $CF_{mix} = X_1 * CF_1 + X_2 * CF_2 + X_3 * CF_3 + \dots + X_i * CF_i$ 

An example of the calculation of the response factors for a composition, is shown in **Table 2**.

<sup>&</sup>lt;sup>7</sup> Thermo Fisher Scientific – Air Quality Instruments – 27 Forge Parkway - Franklin

Component	MW	RF	RF corrector	mass%	mass in 100 g	mass (in 100g)/MW	mol%	RF composition	RF corrector composition
benzene	78,00	0,35	0,23	1,0	1,0	0,01	1,4	0,00	0,00
butane	58,00	0,58	0,32	2,0	2,0	0,03	3,8	0,02	0,01
decane	142,30	0,42	0,88	13,0	13,0	0,09	9,9	0,04	0,09
dodecane	170,00	1,00	0,00	13,0	13,0	0,08	8,3	0,08	0,00
heptane	100,00	0,39	0,28	11,0	11,0	0,11	12,0	0,05	0,03
hexane	86,00	0,43	0,30	11,0	11,0	0,13	13,9	0,06	0,04
nonane	128,00	0,37	0,38	13,0	13,0	0,10	11,1	0,04	0,04
octane	114,00	0,35	0,28	11,0	11,0	0,10	10,5	0,04	0,03
pentane	72,00	0,51	0,32	10,0	10,0	0,14	15,1	0,08	0,05
propane	44,00	0,62	0,21	2,0	2,0	0,05	4,9	0,03	0,01
undecane	156,00	1,00	0,00	13,0	13,0	0,08	9,1	0,09	0,00
Total				100	<u>100</u>	1	<u>100</u>	<u>0,53</u>	<u>0,31</u>

#### Table 2 Example of the calculation of response factors of a stream composition (COMP001)

The measurements of a certain stream (with a certain composition) are performed after entering the calculated response factors for the composition. As such, the TVA 1000B transforms the reading for methane automatically, using the following formula:

$$Y = \frac{AX}{\left(1 + \frac{BX}{10.000 \ ppm}\right)}$$

X represents the measured concentration, while Y represents the "corrected" concentration for the composition measured.

A represents the response factor multiplier at very low concentrations and B represents a change to the response for every percent concentration increase. A and B were determined by Thermo Instruments for a certain number of components.

Thus, for example, a vapour phase mixture of 5% benzene and 95% n-hexane would have a  $CF_{mix}$  (500) of

 $CF_{mix} = 0.05 * 0.35 + 0.95 * 0.43 = 0.42$ 

A reading of 100 would then correspond to 42 ppmv of the total mixture, comprised of 2 ppmv of benzene and 40 ppmv of hexane. These calculations are performed automatically by the software.

During stream determination and database setup, all correct response factors for each chemical compound for the detection devices are being added to the software. Note that only for a selected list of individual components are response factors available; when components are present in a composition for which no response factors are available, default factors are used, or sometimes the choice is made to use response factors of a component from the list resembling chemically the one in question. Based on stream compositions, a stream response factor is calculated and applied to the associated sources. All of these stream response factors are listed and used by onsite technicians to guarantee correct setup of the detection equipment in the field. Due to usage of this list, combined with the P&ID's with the marked streams and stream identification, correct concentration readouts can be assured.

## ANNEX B – REFERENCE TABLES OF OGI CAMERA DETECTION LIMITS

Concawe performed a controlled leak test in spring 2013 at a test facility for LDAR training and studies which enables leaks of different sizes and from different equipment types to be generated. The field detection limit of the FLIR GF 320 camera which was used during the field campaigns was tested. The test was performed according to the EPA-AWP<sup>g</sup>.

The aim of the test was to detect which flow rates (g/h) were visible using the IR camera at 2 meters distance in normal or high-sensitivity mode. Results of that test are given in section 3.2.2.1. In this annex, reference tables of OGI camera detection limits are given. Detection limits are in accordance with the detection limits obtained during the field measurement campaigns and controlled leak study performed by Concawe.

Table 1	Summary of FLIR GasFindRTM MW camera method detection limits and percent
	agreement with a method 21 monitoring device during laboratory testing <sup>9</sup>

	Method Detection Limit (g/hr)		Agreement with Method 21 Monitoring Device	
			Total No. of Tests	
Compound	Minimum	Maximum	Performed	Percent Agreement
1,3-butadiene	1.3	2.7	4	100%
Acetic acid	≤ 0.02	$\leq 4.6^{(b), (c)}$	11	100%
Acrylic acid	0.92	1.2	4	100%
Benzene	0.35	35 <sup>(c)</sup>	12	100%
Methylene chloride	4.9	> 70 <sup>(c)</sup>	No data <sup>(d)</sup>	
Ethylene	0.35	278 <sup>(c)</sup>	8	100%
Methanol	0.28	22 <sup>(c)</sup>	No data <sup>(d)</sup>	
Pentane	≤ 0.28	28 <sup>(c)</sup>	16	100%
Propane	≤ 0.44	13 <sup>(c)</sup>	No	o data <sup>(d)</sup>
Styrene	0.35	0.70	3	100%

(a) Minimum and maximum method detection limits shown were measured at a 0-mph wind speed unless otherwise noted.

(b) Measured at a 2.5-mph wind speed.

(c) Measured at a 5-mph wind speed.

(d) Percent agreement was not evaluated for methylene chloride, methanol, and propane because these compounds have an ionization potential greater than the energy which could be supplied by the Industrial Scientific IBRID MX6 with PID sensor.

<sup>&</sup>lt;sup>8</sup> Controlled Laboratory Sensitivity and Performance Evaluation of Optical Leak Imaging Infrared Cameras for Identifying Alkane, Alkene and Aromatic Compounds

<sup>&</sup>lt;sup>9</sup> Environmental Technology Verification Report, FLIR SYSTEMS, GASFINDIRTM MIDWAVE (MW) CAMERA

Table 2	Summary of controlled laboratory testing detection limits for pure gases for GasFindIR
	cameras <sup>10</sup>

Wind speed	0 mph	2 mph	5 mph
Pure Gas	g/h	g/h	g/h
Butane	0.72	5.8	15.9
Ethane	0.97	5.2	17.8
Methane	3.96	20.8	49
Propane	0.76	9.8	19.1
Ethylene	13.9	53.7	104
Propylene	4.37	15.6	59.8

Table 3

Summary of controlled laboratory testing detection limits<sup>11</sup>

Wind speed	0 mph	2 mph	5 mph		
Compound	g/h	g/h	g/h		
Benzene	>70.1 <sup>B</sup>	>70.1 <sup>B</sup>	>70.1 <sup>B</sup>		
Ethylbenzene	7.6	53.2	>75.9 <sup>B</sup>		
Heptane	3	21	48		
Hexane	2.9	37.6	57.8		
Isoprene	32.8	59.6	>59.6 <sup>B</sup>		
Methanol	16.7	41.7	69.3		
MEK	5.3	60	>70.5 <sup>B</sup>		
MIBK	7.01	24.6	70.1		
Octane	4.36	18.7	62.2		
Pentane	13.8	25.4	45.8		
1-Pentene	14	30.9	47.7		
Toluene	22.6	>75.3 <sup>B</sup>	>75.3 <sup>B</sup>		
Xylene	15.1	52.8	>75.3 <sup>B</sup>		

<sup>B</sup> Note that the detection limits with a greater than symbol mean that the detection limit for this technology was greater than could be reproduced in the laboratory. This does not mean that the camera cannot see this compound at any level, just that a leak of sufficient size could not be created in a safe and measurable manner in the laboratory setting.

<sup>&</sup>lt;sup>10</sup> Controlled Laboratory Sensitivity and Performance Evaluation of Optical Leak Imaging Infrared Cameras for Identifying Alkane, Alkene and Aromatic Compounds

<sup>&</sup>lt;sup>11</sup> Controlled Laboratory Sensitivity and Performance Evaluation of Optical Leak Imaging Infrared Cameras for Identifying Alkane, Alkene and Aromatic Compounds

# ANNEX C – BAGGING METHODS: VALIDATION IN A CONTROLLED EXPERIMENT AND FIELD COMPARISON

In all campaigns, high flow sampling (HFS) was selected as the method of choice for independent leak quantification. HFS is not a referenced method for bagging, whereas vacuum bagging is referenced by EPA-435/R-95-017. Both methods were assessed by:

- Performing a controlled experiment in test facilities (where a known leak rate was generated and measured independently by each bagging method)
- Comparing their results in the field (side by side bagging by each method was performed on a selected number of leaking components in Site 2 during the first campaign)

### Bagging comparison – controlled experiment

The two bagging methods (vacuum bagging and high flow sampling) that were used to estimate emission leak rates during the field campaigns were compared and the accuracy of the two methods investigated.

The comparison of the methods was performed by generating a known leak rate of a gaseous stream and comparing this value to the mass emission values calculated using the bagging methods.

#### 1.1. INSTALLATION AND METHODS

For this study the LDAR-installation was used for the controlled generation of the volatile organic gas leaks. In this case for each experiment one leak at a time was generated, with propane (purity 99.9%) being used as the leak material. A mass flow controller (MFC) was used to control and determine the leak mass flow rate. In total, five leak rates were tested in the range of 12 g/h up to 2000 g/h. All leak rates were sampled using both bagging methods.

All the tests were performed on the same equipment piece, in this case a valve. The valve that was chosen had two leaking points, at the stem and at the flange.

<u>The vacuum bagging method</u> was performed according to the description provided in section 4.1."EPA – vacuum method and blow-through method". More details can be found in reference 5: "Protocol for Equipment Leak Emissions Estimates. For each leak rate, two gas samples were collected in sample bags at the outlet of the vacuum pump and analysed in the laboratory with a FID analyser.

<u>The high flow sampling</u> method was performed according the description given in Annex D and section 4.2 "High Flow Sampling (HFS)". In this method the leak flow rate, diluted with air, was measured by the high flow sampling device (manufactured by Bacharach whereas the hydrocarbon concentration was measured at the exhaust with a Toxic Vapor Analyser (TVA) in the FID mode. In the controlled experiment, hydrocarbon concentration measurements at the exhaust of the HFS were also performed with a propane calibrated FID. As part of the method quality check (see also Annex D) the sampling was conducted at both a"high flow" rate (225 l/min) and at lower flow rate (25% of the high flow rate). This was also done in all field measurements.

The calculation methods and calibration procedures are described in more details in Annex E.

## 1.2. <u>RESULTS</u>

#### Vacuum bagging

Results of the vacuum bagging and their deviations from the known leak rate are given in **Table 1**, columns 2 and 3. In column 5 are given the leak rates estimated using the measured leak concentration and applying Method 21. More detailed results are given in **Annex E**.

TOC generated (g/h)	Vacuum bagging TOC calculated (g/h)	Deviation Vacuum bagging (%)	Screening value ppmv	TOC calculated with EPA method 21 formula (g/h)	Deviation Method 21 (%)
11.68	14.7	26%	6,964	5.1*	-56%
48.59	58.7	21%	>20,064	>11.24**	-77%
209	214.3	3%	>100,000 Pegged	140	-33%
1027.8	1,004.1	-2%	>100,000 Pegged	140	-86%
2053.7	2,012.1	-2%	>100,000 Pegged	140	-93%

Table 1         Results - vacuum bagging method and method EPA 21 ca	alculations
--	-------------

\*Average of the screenings performed before and after the test at 11.68 g/h.

\*\*The recorded screening values were >10,000 ppmv methane and at this point the response factors (here for propane) start to deviate. More explanation is given in section 1.3 below.

#### High Flow Sampling

For the HFS TVA experiment the lowest leak rate (11.68 g/h) was measured with a TVA without dilution probe. The leak rates of 48.59 g/h and 209.0 g/h were measured using the TVA dilution probe. In retrospect, the 48.59 g/h could have been measured without the dilution probe.

The concentrations were measured in ppmv methane (as TVA was calibrated with methane) and response factors were applied to recalculate these concentrations to ppmv propane. The application of response factors is described in more details in Annex A. The propane concentrations were also measured by an independent FID and the values were also used to calculate the mass flow rate. The deviation between this calculation and the generated leak rate is listed in the column "propane calibrated FID" in **Table 2**).
The results of the HFS and the deviations from the known leak rates for the different leak rates are given **Table 2**.

TOC generated (g/h)	Flow rate HFS (I/min)	Concentration (ppmv) – measured with TVA-1000B at the exhaust of the HFS	TOC (g/h) Calculated with recalculated TVA-1000B reading at the exhaust of the HFS	Deviation (%) Calculated with recalculated TVA-1000B reading	TOC (g/h) Calculated using propane calibrated FID	Deviation (%) Propane calibrated FID
11 60	227	800	12.0	3%	12.9	10%
11.00	165	1,100	11.9	2%	13.0	11%
19 50	212	6,000*	48	0%	48.5	0%
40.39	180	7,020*	48	-1%	48.0	-1%
200.0	155	80,000*	403	93%	181.2	-13%
209.0	225	36,000*	285	36%	206.8	-1%
1007.0	202	Flame out	-	-	1156.5	13%
1027.0	176	Flame out	-	-	1077.2	5%
2052 7	224	Flame out	-	-	2144.7	4%
2003.7	185	Flame out	-	-	2126.4	4%

Table 2HFS - Leak calculations

\*Note: Dilution probe used; dilution factor was 5.6 based on the calibration method of the dilution probe

# 1.3 CONTROLLED LEAK TVA-1000B STUDY

In the study described above, the results obtained using the TVA-1000B and the HFS showed a higher deviation (versus the generated leaks) than the deviation of the laboratory propane calibrated FID. Concawe investigated the reason for this deviation in a subsequent study.

During that study, a literature survey was done first, related to the working principle of the TVA-1000B and the use of response factors provided by the manufacturer. Secondly, it was studied in a controlled leak test whether the deviation between HFS and the known leak rate, observed in the previous study, could be the result of the use of the methane calibrated TVA-1000B and the recalculation using the response factors, or other factors like the dilution probe, linearity of the equipment, etc.

Portable FIDs are usually calibrated with methane at zero and 10,000 ppmv (single point calibration). The response of a methane calibrated FID is linear in a broad range (up to 10,000), but it is known that the response is not linear at higher concentrations.

Accuracy and linearity of the TVA-1000B for four different components (methane, propane, toluene and propylene) were tested. For methane and propane a total of 6 concentrations were generated in the range of 0 - 50,000 ppmv. For toluene and propylene 5 concentrations were generated in the range of 0 - 50,000 ppmv.

10,000 ppmv. The ranges of the generated concentrations were limited due to safety and technical reasons.

Every measurement with the TVA-1000B was performed with and without a dilution probe. Comparing the undiluted and diluted measurements should give an idea of the possible advantages or errors introduced using the dilution probe. All measurements were performed using two portable methane calibrated TVA 1000B FIDs. This was done to eliminate possible errors related to malfunction or errors of a single piece of equipment. The dilution factor chosen in this study was 8 (from range of 1 to 10) for all the measurements.

#### Methane calibrated FID measuring propane

The results showed that the response factors for the methane calibrated TVA-1000B are valid up to a screening value of approximately 30,000 ppmv. With the dilution probe, it is possible to extend the measurement up to a screening value of approximately 50,000 ppmv (due to dilution the screening value is "lowered" back to the linear area of the response curve). The results are illustrated in **Table 3** and **Figure 1**.

	Without	dilution pro	obe	With dilution probe (factor 8)						
Known propane leak	TVA 1000B measurement	RF correction	Deviation	TVA 1000B measurement	RF correction	No dilution	Deviation			
ppmv	ppmv methane eq.	ppmv propane	%	ppmv methane eq. diluted	ppmv propane diluted	ppmv propane	%			
1042	1194	722	-31%	144	89	712	32%			
5285	8350	4405	-17%	810	494	3950	25%			
10,319	23,600	9783	-6%	1782	1065	8520	-18%			
20,019	91,500	19,418	-3%	4060	2319	18,556	7%			
30,073	241,000	24,653	-18%	7160	3859	30,872	-3%			
48,955	Flame out	-		14,000	6708	53,663	-10%			

#### **Table 3**Methane calibrated TVA1000B (FID) with propane leak



# *Figure 1* Methane calibrated TVA 1000B (FID) with propane leak

The deviation observed for the TVA-1000B in the linear range is in the same order of magnitude as the deviation observed when the propane leak concentration was measured at the exhaust of the HFS (**Table 2**, lines 1-2-3-4-6). The first concentration measurement of the 200 g/h leak rate (**Table 2**, line 5) was above 50,000 ppmv methane eq. and the deviation is higher.

Methane calibrated FID measuring other gases

Similar results to propane were observed with methane, propylene and toluene.

Propane calibrated FID

The study indicated that propane is not a good gas for calibrating the TVA-B because the linearity range is low (only up to 30,000 with the dilution probe).

All details of this study are available in reference 18 (which can be consulted on request).

# 1.4 CONCLUSIONS OF CONTROLLED EXPERIMENTS

**Vacuum Bagging method -** the calculated leak rates show small deviations for the three highest leak rates: 3%, -2% and -2% respectively for leak rates of 209.0 g/h, 1027.8 g/h and 2053.7 g/h. For these three experiments, the leaks were so high that close to saturation concentration was achieved in the bag (propane concentration above 90%). This situation rarely happens in the field because leak rates are much smaller and there is always some air in the bag. These three concentrations, therefore, could be

considered to represent one experiment. For the two smallest leak rates of 11.68 g/h and 48.59 g/h the deviation compared to the generated leak was higher; respectively 26% and 21%.

Based on the results of the controlled experiment, it can be concluded that the vacuum bagging method is applicable for sampling the entire tested range of leak rates (12 to 2000 g/h). The highest deviation between generated and measured leak rates in the tests was 26%.

**High Flow Sampling method -** no leak rate calculation was possible for the two highest generated leaks (1000 and 2000 g/h) because the TVA-1000B flamed out at the exhaust of the HFS (see **Table 2**). In the field tests, this situation was never observed, meaning that the leaks bagged with HFS were lower than 1000 g/h.

The control measurement performed with the propane-calibrated FID (laboratory instrument) at the exhaust of the HFS showed lower deviations for all generated leak rates (between -13 and +13%, with an average of 3%). This indicates that the deviation in the leak rate estimation is not due to the volumetric flow estimation by the high flow sampler but is likely to be caused by the concentration inaccuracy. The additional tests (detailed in section 1.3) exploring the inaccuracies introduced by the TVA-1000B, the response factors and the dilution probe confirm this hypothesis.

The TVA-1000B is a portable FID instrument, suitable to be used in the field, requiring daily calibration with methane. Its linear range is limited to 30,000 ppmv and can be extended to 50,000 ppmv when combined with the dilution probe. For measuring other gases, the response factors provided by the manufacturer can be used but their linearity is limited to measurements below 10,000 ppmv (with or without dilution probe).

Based on the results of the controlled experiment, it can be concluded that the HFS bagging method is only applicable in the linearity range of the TVA-1000B instrument. This range does not exceed 200 g/h propane as illustrated in **Tables 2** and **3**, considering the following conditions, applied both in the controlled experiment and in the field:

- most of the HFS flow measurements were made at 225 l/min (maximum flow of the high flow sampler) and 169 l/min (25% lower than max flow rate)
- the highest TVA-1000B concentration measured in the linear range, using the dilution probe, is 50,000 ppmv
- the dilution probe used by the contractor performing the HFS had a dilution rate of 5.6
- the undiluted propane concentration was 10,932 ppmv (using the TVA1000-B response factor for propane)
- Assuming a temperature of 15C, the propane mass rates were respectively 267 and 200 g/h

For other gases or mixtures, the maximum range will be slightly different, depending on the molecular weight and the response factor (and the ambient temperature).. For simplicity, all field leaks for which a HFS concentration was measured above the linearity range of the TVA-1000B combined with the dilution probe were assigned a fixed flow rate of 200 g/h.

For all other (smaller) leak rates the accuracy of the HFS is comparable to that of vacuum bagging (< 36%, as shown in **Table 2**)

#### Important remark

No leak rates below 12 g/h could be generated in the controlled experiment due to equipment limitation, and therefore the accuracy of the two bagging methods could not be evaluated for the very low leak rates.

This was not considered an issue for the study because the smallest leak rates do not significantly contribute to the mass emission and can be considered as one group in the leak rate analysis.

# 2. BAGGING COMPARISON - FIELD STUDY RESULTS

Vacuum bagging was performed on a limited number of leaking components in Site 2 during the first field campaign and was compared to HFS. **Table 5** shows the leak rate estimated by vacuum bagging (third column), by HFS (fourth column) and the deviation between the two values.

The data illustrated in **Table 5** show a larger deviation between the two bagging methods when applied in the field than their expected inaccuracy based on the controlled experiments. The following factors may explain the larger deviations observed in the field:

- Vacuum Bagging and HFS took place during the same week but could not always be performed one after the other; when separated by a timespan of several hours or days, the leak rate can have changed.
- While HFS was performed by the same team and with the same equipment in the field and in the controlled experiment, it was not the case for vacuum bagging.
- HFS is a more robust methodology for field application:
  - During the field campaign in Site 1, HFS was repeated twice on most of the 74 leaks bagged and its repeatability was very good;
  - Bagging is easier as some air is allowed to enter the bag;
  - No need to establish equilibrium concentration in the bag as the leak is "sucked" by the HFS at higher flow rate;
- Vacuum bagging performed in the field is expected to be less accurate than in the controlled experiment, particularly for the lowest leak rates:
  - For small leaks, it takes time to establish equilibrium concentration in the bag, due to the low established flow rate. In the controlled experiment, at least 30 minutes were needed for the lowest flow rate (but a larger bag was used than in the field). In the field, all vacuum bagging measurements were done within a few minutes. A smaller bag was used (requiring less time to reach the equilibrium concentration) but the varying concentrations recorded at the exhaust of the sampling train showed that equilibrium was not necessarily achieved.
  - For some leaks, the concentration in the bag was low, while the measurements with the TVA-1000B at the exhaust of the vacuum bagging train indicated a high VOC concentration; in such situation, vacuum Bagging is likely to have under-estimated the leak rate.

HFS ID	Sample ID	VB (g/h)	HFS (g/h)	Deviation VB - HFS/(aver age HFS,VB)	Note
34	001 A/B	18	18	0%	
43*	002 A/B	104	200	-63%	Dilution probe was used
33	003 A/B	11.2	2	139%	Measurement done on a different day
49	004 A/B	6.4	53	-157%	?
53	006 A/B	10.5	65	-144%	Pegged value at vacuum bagging (VB) exhaust contradicts low VB TOC
76	009 A/B	1.5	3	-67%	
77	010 A/B	0.2	2	-164%	Measurement done at a different time of the day; very low leak rate during vacuum bagging
70	011 A/B	2.4	1	82%	
67	012 A/B	20.4	11	60%	
68	013 A/B	1.4	2	-35%	
69	014 A/B	7.8	44	-140%	?
71*	015 A/B	48.8	200	-122%	Dilution probe was used
72	016 A/B	0.1	NA		
78	017 A/B	5.2	4	26%	
75	018 A/B	4.4	8	-58%	
56	019 A/B	8.9	29	-106%	Measurement done on a different day
61	020 A/B	8.3	30	-113%	Measurement done on a different day
60	021 A/B	31.2	141	-128%	Measurement done on a different day. Higher inaccuracy for HFS. >10000 ppmv methane, without dilution probe, response factor not valid anymore
NA	022 A/B	1.6		100%	
65	023 A/B	4	8	-67%	
66.2	024 A/B	1	9	-160%	Measurement done on a different day

# Table 4 Leak rate estimation by vacuum bagging and HFS

\*Dilution probe used

In most of the cases, the mass leak rate determination by HFS was higher than vacuum bagging and therefore provides a conservative determination of the mass leak rate. On this basis, HFS was considered acceptable as the independent leak quantification method for the Concawe study.

For the leak rates determined by HFS between 0.1 - 200 g/h, the values were used as such in the graphs. For the leak rates measured with the dilution probe and exceeding the validity range of HFS, a fixed mass emission of 200 g/h was assigned for simplicity. This approach is similar to the use of "pegged values" in Method 21.

# ANNEX D – HIGH FLOW SAMPLING PROCEDURE

#### Introduction

The high flow sampler is a portable, intrinsically safe, battery-powered instrument originally designed to determine the rate of gas leakage around various pipe fittings, valve packings and compressor seals found in natural gas transmission, storage and compressor facilities. This is accomplished by sampling at a large flow rate (between 5 and 10.5 scfm) to completely capture all the gas leaking from the component along with a certain amount of air. By accurately measuring the flow rate of the sampling stream and the natural gas concentration within that stream, the gas leak rate can be calculated.

#### **1.1. OPERATION PRINCIPLE**

The High Flow Sampler uses the principle of "bagging" to measure the flow. This is the most accurate method to estimate emissions according to EPA.

The bag was closed up, air was allowed to flow into the bag to reduce the vacuum in the enclosure. However, a slight vacuum was maintained in the bag. Then, the high flow sampler's main sampling hose was attached to the bag.

A TVA (FID) was placed at the exhaust of the high flow sampler to log the concentration in ppmv during approx. 3 minutes. From this log the average concentrations were calculated. These ppmv values in combination with the flow are used to calculate the losses, according to the following formula:

$$Loss\left(\frac{g}{h}\right) = \frac{Concentration * MW * Flow}{R * Temp} * 10^{-3}$$

Where:

Loss = Average or Maximum loss (g/h) Concentration = Actual Concentration (ppmv) (average of the concentration log) MW = Molecular Weight (g/mole) Flow = Flow (m<sup>3</sup>/h) R = Gas Constant (0.0820578 L\*atm\*K<sup>-1\*</sup>mol<sup>-1</sup>) Temp = Temperature (K)

For example, leak number 45 had a composition with molecular weight 145.31 g/mole. The flow recorded by the HFS was 138 l/min (8.28 m<sup>3</sup>/h). The temperature at the exhaust of the HFS was 30.3°C and the average concentration (calculated from the log recorded by the TVA) was 1,006 ppmv. As such, the emission calculated according to equation above was: 48.6 g/h or 426 kg/yr.

The concentration was measured at two different flows (maximum and medium flow, referring to the flow range of the device). This gave rise to two parallel loss calculations. However, the resulting losses should not deviate by no more than 10% for optimal results to be obtained.

To check the precision of the method, the sampling was conducted at both a "high flow" rate (225 l/min) and a lower flow rate (25% of the high flow rate). The measurement results had to agree with 10% for the test to be considered valid. The following formula has been used for the HFS quality check:

 $\frac{(flow1 \times conc \ 1) - (flow \ 2 \times conc \ 2)}{(flow \ 1 \times conc \ 1)} < 10\%$ 

## High Flow Sampling experimental protocol – Field measurement campaigns

During the field measurement campaigns the high flow sampling procedure complied with the following points, based on EPA-453/R-95-017:

- 1. The composition of material in the designated equipment component and the operating conditions of the component were available from the database
- 2. A screening value of the leaking point was recorded with the TVA 1000B
- 3. A bag was cut from appropriate material that easily fit over the equipment component.
- 4. The VOC concentration was observed at the vacuum pump exhaust with the TVA 1000B. It was made sure that the concentration stayed below the lower explosive limit. The concentration was logged during approximately 3 minutes. Afterwards, the average concentration of the log was calculated; this average concentration was used in the emission calculation. Normally, the concentration profile was quite stable. However, on a few occasions, the concentration profile showed deviations.
- 5. The concentration profile was only taken once; no repeats were performed (except for some leaks during Campaign I, when the deviation on the loss at high vs medium flow was > 10%)
- 6. When higher concentrations were encountered, the dilution probe was used.
- 7. A quality check of the high flow sampling was carried out using two different flow sampling speeds and recording the associated VOC concentration; the leak rate calculated by both modes should not differ by more than 10%. During Campaign I, a few leaks for which the deviation was > 10% were repeated. In general, when the deviation was > 10%, it was included in the remark for the leak in question, but no changes were made to the setup (as the calculation could only be performed afterwards). The data were not discarded.
- 8. The camera was used to check the bag on a few occasions; these examples showed that the leak was fully captured. However, the time was limited and the camera was needed to perform the OGI screening; therefore it was difficult to use the camera every time a leak was bagged. For this reason, it was agreed to check the leak capture by the bag by screening with the TVA in the vicinity of the bagged component. These screenings showed that the leak capture was successful on all occasions.
- 9. The bag was purged after use till recording < 10 ppmv VOC concentration
- **10.** Duct tape was used for tightening the bag

# Specifications of the High Flow Sampler

Measured Values	Sampling flow rate
	Background gas concentration
	Sample gas concentration
	Battery voltage
Calculated Values	Leak concentration corrected for background gas
	level
	Leak rate
	Percent difference between leak rate
	measurements #1 and #2
Measurable Leak Rate	0.05 to 8.00 SCFM (1.42 to 226 LPM)
	0.05 to 6.00 SCFM (1.42 to 170 LPM)
Accuracy of Calculated	
Leak rate	±10% of reading
Temperature	
Operating	0 to 50 °C (32 to 122 °F)
Storage	-40 to 60 °C (-40 to 140 °F)
Humidity	5 to 95% RH (non-condensing)
Sampling Flow Rate	
Maximum	10.5 SCFM (297 LPM) at full battery charge
<b>Operating Flow Points</b>	Initial flow approx. 10 SCFM (283 LPM)
	Second flow approx. 8 SCFM (226 LPM)
	(The second flow rate is 25% of the initial flow)
Measurement Method	Differential pressure across restriction
Accuracy	±5% of reading
Natural Gas Sensor	
Detection Method	Catalytic oxidation / Thermal conductivity
Range	
Catalytic oxidation	0 to 5% by volume methane
Thermal conductivity	5 to 100% by volume methane
	±5% of reading or 0.02 % methane, whichever is
Accuracy	greater

# ANNEX E – CONTROLLED BAGGING STUDY – CALIBRATIONS, CALCULATIONS AND ADDITIONAL RESULTS OF THE STUDY

A study to compare the two bagging methods which were used to estimate emission leak rates was performed in a leak detection and repair (LDAR) installation (a test facility for LDAR training and studies which permits leaks of different sizes, from different equipment types, to be generated) in Spring 2014. These two methods are the high flow sampling method (HFS) and the vacuum bagging method which is described in EPA-453/R-95-017 [8].

The comparison of the methods was be performed by generating a known leak rate (in g/h) of a gaseous stream and comparing this value to the calculated mass emission by both bagging methods.

For this study the LDAR installation was used for the controlled generation of the volatile organic gas leaks. In this case for each experiment one leak at a time was generated. Propane was used to generate the leaks and a valve (**Figure 1**) used as a leaking point. The valve that was chosen had two leak points; at the stem and at the flange. The flow rate and concentration of the generated flow were alternated in order to have a comparison at different levels of "leakage". In total five leak rates were tested in the range of 12 g/h up to 2,000 g/h. All leak rates were sampled using both bagging methods.

*Figure 1* Leaking equipment piece



For the high flow sampling set up, the concentrations were measured at the outlet of the HFS. The measurement was performed with the TVA-1000B, or with the internal sensors of the HFS when the concentrations at the exhaust exceeded the measuring range of the TVA-1000B. As a control, an additional propane calibrated FID was used at the exhaust of the HFS.

# 1. CALIBRATIONS - INSTRUMENTS CALIBRATION, PRE-TEST AND POST-TEST VERIFICATION

Leak generation – Mass Flow Controller (MFC) Calibration

**Table 1** gives an overview of all calibration data of the MFCs used for the leak generation. Every MFC set point used for the leak generation was calibrated the day before the start of first test and calibrated again after finishing the last test.

The calibration data in

**table 1** are the data of the calibration before the start of the first test. The calibrations performed after all tests were finished were all within 0.2% of the first calibration results.

Mass flow controller	Set point (% of max range)	Volume of the mercury piston (ml)	Time (s)	Temp (°C)	Pressure (mbar)	Calibrated flow (Nml/min)	Calibrated leak rate (g/h)
	4%	75	42.96	22.5	1,013.8	96.8	11.68
MFC MIE-ILU-772	16%	200	27.51	22.8	1,014.0	402.9	48.59
	70%	1,980	63.4	22.5	1,014.3	1,732.9	209.0
MFC MIE-ILU-688	52%	6,000	39.15	22.6	1,016.8	8,522.1	1,027.8
MFC MIE-ILU-219	49%	6,000	39.21	22.7	1,016.8	8,506.2	1,025.9

#### Table 1 Calibration data leak generation before the start of the test

Calibrated leak rates

Table 2 gives the difference between the proposed and received leak rates.

 Table 2
 Proposed and actual leak rates during the measurements

Proposed leak rate (g/h)	Calibrated (actual) leak rate(g/h)
12	11.68
50	48.59
200	209.0
1000	1,027.8
2000	2,053.7

#### Daily TVA calibration

At the start of each day the TVA-1000B was calibrated. The TVA was calibrated using a 10,000 ppmv methane in air calibration gas and a 0% methane in air. The calibration linearity was checked with a control gas of 500 ppmv methane in air.

#### Daily system leak check

At the beginning of each testing day the bagging train used in the Vacuum Bagging method was checked for leaks. The leak check was performed by closing the suction tube and waiting until the dry gas meter reading drops to zero.

All leak checks passed the control.

#### Calibration of the two high flow sampler gas sensors (catalytic oxidation and thermal conductivity)

At the start of the first and the second testing day, the HFS was calibrated using a 2.5% methane in air and a 100% methane calibration gas. Manufacturer's instructions were followed and calibration appeared to be successful. However, the subsequent measurements showed that there was something wrong:

- In one case, there was no reading at all
- In the second case the reading did not pass the QA/QC test suggested by the manufacturer

Consequently, these measurements are reported but cannot be used to compare the bagging methods.

#### FID calibration (direct measurement and sample bag analysis)

The two FID's (JUM 3-300-A) used for the direct measurement at the exhaust of the high flow sampler and for the analysis of the diluted sample bags (in the lab) were calibrated with ISO 17025 certified calibration gases (calibration gas concentrations of 90 ppmv propane or 900 ppmv propane). The calibration was performed at the beginning and end of each day.

# 2. CALCULATIONS METHODS

#### Vacuum bagging

For the Vacuum bagging, the TOC was calculated using the following formula:

Leak Rate 
$$(g/h) = \frac{9,63 \times 10^{-10} (Q)(MW)(GC) \times 1000}{T + 273,15}$$

With:

Q = Flow rate out of Bag (l/min) MW = Molecular weight of molecular compound (propane= 44.1 g/mole) GC = Sample bag organic compound concentration (ppmv) P = Pressure at the dry gas meter (mmHg) T = Temperature at the dry gas meter (°C)

In all experiments the vacuum in the bagging train was kept constant. All pressures were in the range of - 0.23 mmHg up to -0.29 mmHg.

The deviation of the calculated TOC in comparison to the generated TOC is calculated as follows:

TOC(calculated) - TOC(generated) TOC(generated)

# Method 21

Using the results of the initial TVA screening, the leak rate was also calculated using the EPA method 21 correlation method. The leak rates in g/h were calculated using the formula for valves in Table 2-10 Petroleum industry leak rate/screening value correlations in EPA method 21:

Leak rate  $(kg/h) = 2,29 * 10^{-6} * ScreeningValue^{0,746}$ 

For the three biggest leaks the TVA screening gave 'Flame out', therefore the pegged value for valves (0.14 kg/h) was used.

## High flow sampler (HFS)

The volumetric flow rate of the high flow sampler pump is calculated from the pressure differential across a critical orifice. The calibration is done by the manufacturer and it cannot be changed by the user. The leak rate is then calculated as the product of the concentration and flow rate, corrected to standard conditions.

To check the precision of the method, the sampling was conducted at both a "high flow" rate (225 l/min) and a lower flow rate (25% of the high flow rate). The measurement results must agree with 10% for the test to be considered valid. The following formula have been used for the HFS quality check.

$$\frac{(flow1 \times conc \ 1) - (flow \ 2 \times conc \ 2)}{(flow \ 1 \times conc \ 1)} < 10\%$$

The TVA readings were recalculated by using the response factors (See Annex A). For the calculation of the leak rate (TOC) with the recalculated TVA reading and HFS measurement, the following formula is used:

Leak rate 
$$(g/h) = \frac{Concentration \times MW \times Flow}{R \times Temp} \times 10^{-3}$$

With:

MW Flow

R

Concentration = Actual Concentration (after recalculation) (ppmv) = Molecular Weight (propane= 44.1 g/mole) = Flow (m<sup>3</sup>/h) = Gas Constant (0.0820578 L\*atm\*K-1\*mol-1 Temp = Temperature (K)

For the concentrations measured with the TVA-1000B dilution probe (leak rates 48.59 g/h and 209.0 g/h) the recalculation was done using the diluted measurement result. The multiplication with the dilution factor (factor = 10) was done after the recalculation.

As a control of the method, the hydrocarbon concentration of the diluted mixture was measured using a FID analyser. This FID analyser (Type: JUM 3-300A) was calibrated with ISO 17025 certified calibration gas (propane). The hydrocarbon concentration was measured directly at the exhaust of the high flow sampler when the concentration was below 1%. When higher concentrations were measured the gas at the exhaust was collected using a sample bag and analysed in the lab with a FID analyser. The TOC calculated with this additional propane calibrated FID measurement result was calculated with the formula used for the vacuum bagging method mentioned above.

# 3. ADDITIONAL RESULTS

The following tables summarize the results which are not presented in the main body of the report.

14510 0	The bamp	ing data			
TOC generated (g/h)	Date and time	Initial TVA screening (ppmv CH4)	Flow rate HFS (I/min)	Barometric pressure (mmHg)	Temperature at outlet HFS (°C)
11.68	3/04/2014	14 700 (*)	227	750	24.3
11.00	15h00	14,700()	165	750	23.8
19 50	3/04/2014	>101 000	212	745.6	17.5
40.09	10h20	>101,000	180	745.6	17.5
200.0	2/04/2014	Elamo out	155	752.9	22.2
209.0	16h35	Fiame out	225	752.9	22.5
1027.9	3/04/2014	Elamo out	202	750.6	27.4
1027.0	14h20	Fiame Out	176	750.6	25.3
2052 7	3/04/2014	Elamo out	224	749.8	27.1
2003.7	13h15	Fiame out	185	749.8	27.1
	A				

Table 3	HFS -	sampling	data

(\*) Average of the screenings performed before and after the test at 11.68 g/h

TOC generated (g/h)	Flow rate HFS (I/min)	TVA measurement (ppmv CH4) with dilution	Leak conc ppmv CH4)	Recalculation (ppmv C3H8) (A=0.62, B=0.21) $\frac{AX}{\left(1+\frac{BX}{10000}\right)}$	Average concentration measured with propane calibrated FID (ppmv C3H8)
11 60	227	-	800	488	530
11.00	165	-	1,100	667	735
19 50	212	600	6,000*	3,674	2,100
40.09	180	702	7,020*	4,289	2,450
200.0	155	8,000	80,000*	42,466	10,800
209.0	225	3,600	36,000*	20,751	8,500
1027.9	202	-	-	-	53,987
1027.0	176	-	-	-	57,311
2053 7	224	-	-	-	90,287
2000.7	185	-	-	-	108,391

\*Note: Dilution probe used

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# Table 5All vacuum bagging results

TOC generated (g/h)	Date and time	barometric pressure (mmHg)	Dry gasmeter temperature (°C)	Initial TVA screening (ppmv CH4)	Flow rate bagging train (I/min)	Vacuum train pressure (mmHg)	Average TOC concentration (bag 1 + bag 2) (ppmv C3H8)	TOC calculated (g/h)	Deviation (%)
11.68	3/04/2014 15h30	750.0	24.8	14,700 (*)	0.697	-0.263	197,329	14.7	26%
48.59	3/04/2014 9h50	745.6	18	>101,000	0.909	-0.240	593,616	58.7	21%
209.0	2/04/2014 14h55	752.9	27.1	Flame out	2.136	-0.285	942,457	214.3	3%
1027.8	3/04/2014 13h55	750.6	28.3	Flame out	10.354	-0.248	917,143	1004.1	-2%
2053.7	3/04/2014 13h40	749.8	27.9	Flame out	19.730	-0.233	964,251	2012.1	-2%

(\*) Average of the screenings performed before and after the test at 11.68 g/h

Table 6 All HFS results

	Sampling data						Measured at the exhaust of the HFS				Leak calculation (g/h)				Deviations			HFS Quality check								
TOC generat ed (g/h)	Date and time	Initial TVA screenin g (ppmv CH4)	Recalculation (ppmv C3H8) (A=0,62, B=0,21) $\frac{AA}{\left(1+\frac{BX}{10000}\right)}$	Flow rate HFS (I/min )	Temp. outlet HFS (°C)	TVA conc (ppmv CH4)	TVA conc (ppmv CH4) with dilution	Recalc. (ppmv C3H8) (A=0.6 2 / B=0.21 ) x (1 + <u>5x</u> (1 + <u>5x</u> )	HFS conc. (ppmv CH4)	Average conc. measure d with propane calibrate d FID (ppmv C3H8)	TOC (g/h) Calculate d with recalcula ted TVA reading	TOC (g/h) Calculat ed with HFS reading using formula	TOC (g/h) Calculat ed with HFS reading using 'High flow QA check' table	TOC (g/h) Calculat ed using VB- formula with propane calibrate d FID - reading	TVA (%)	HFS (%)	Prop ane calibr ated FID (%)	HFS leak measu rement	TVA leak measu rement							
11.68	3/04/2014	14,700	6964	227	24.3	800	-	488	1300	530	12.0	32.0	25.7	12.9	3%	174%	10%	- 27%	-1%							
	151100			165	23.8	1100	-	667	1300	735	11.9	23.3	18.9	13.0	2%	100%	11%									
48 59	3/04/2014 >101.000	>101 000	>20.064	212	17.5	3360	600	2057	-	2100	48.4	-	-	48.5	0%	-	0%	_	1%							
40.00	10h20	2101,000	7,000 220,004	180	17.5	3931	702	2402	-	2450	48.0	-	-	48.0	-1%	-	-1%		170							
200.0	2/04/2014 Flame	2/04/2014 Flame	2/04/2014 Flame	2/04/2014 Flame	2/04/2014 Flame	2/04/2014 Flame	2/04/2014 Flame	2/04/2014 Flar	2/04/2014 Flame		155	22.2	44800	8000	23781	-	10,800	403	-	-	181.2	93%	-	-13%		20%
209.0	16h35	out	-	225	22.5	20160	3600	11621	-	8500	285	-	-	206.8	36%	-	-1%	-	2970							
1007.9	3/04/2014	Flame		202	27.4	-	-	-	44,400	53,987	-	963	784	1156.5		-6%	13%	160/								
1027.0	14h20	out	-	176	25.3	-	-	-	43,000	57,311	-	818	664	1077.2		-20%	5%	10%	-							
		_		224	27.1	-	-	-	53,800	90,287	-	1295	1050	2144.7		-37%	4%									
2053.7 3/04/2014 13h15	3/04/2014 13h15	Flame out	-	185	27.1	-	-	-	44,500	108,391	-	884	721	2126.4		-57%	4%	32%	-							

\*Note: Dilution probe was used with adjusted dilution factor of 10. However, based on the follow up study it was noticed that the adjusted dilution factor of 10 was in reality 5.6 due to uncertain calibration procedure. Dilution factor of 5.6 is used to calculate these results,

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