REPORT

A4.1.2:

Maximum admissible leaks in hydrogen and hydrogen-enriched natural gas pipelines

This report discusses the maximum admissible leaks in hydrogen and hydrogen-enriched natural gas pipelines and the existing metrology requirements and infrastructure for the calibration and traceability of portable hydrogen leak detectors It was written as part of activity 4.1.2 from the EMPIR Metrology for Decarbonising the Gas Grid project, a three year European project commenced on 1st June 2021. For more details about this project please visit www.decarbgrid.eu.

This report was written by:

    Orlando Teodoro, UNL, Nova School of Science and Technology (odt@fct.unl.pt)

Partners: NPL, Enagás, Air Liquide

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1. Introduction

This report aims to review the requirements and existing metrology infrastructure for the calibration and traceability of portable hydrogen leak detectors. It provides guidance to gas operators on how they can ensure traceability when detecting and measuring gas leaks. It will also review common tightness criteria in different scenarios and discuss what extra metrological requirements will bring the use of hydrogen. The ultimate objective is to assess how the metrology infrastructure must respond to these new requirements to ensure adequate traceability of all measured quantities.

Most of this report is focused on what is normally described as 'small leaks' — small but not negligible — so that required tightness is compromised. Thus, in this context, maximum admissible leak is defined as the lowest emission, which may be found in a leak test, that triggers an action — minor leaks are considered negligible, all other are recorded and proper remediation actions are planned. For example, small leaks may be repaired at a convenient opportunity, or just monitored. Larger leaks will certainly be quickly repaired. Leaks in open field may have less strict requirements than others equivalent in metropolitan areas. No matter the action, such leakage should be properly measured, with traceability to SI standards, as in any other measurement.

The goal of this report is to discuss the available documents to anticipate for which range of leakage should the metrology infrastructure provide calibration services. Maximum admissible leaks for the hydrogen and hydrogen-enriched natural gas grid should be established by regulators taking into account safety, environmental, economic or any other relevant considerations. Awareness of maximum admissible leaks is vital to properly plan leak detection campaigns — to select suitable equipment and techniques for the lowest significant leakage, to write the appropriate procedures, to provide adequate training for the technical personnel, to evaluate the cost, etc.

However, before this review, we need to understand which is the relation between the quantities measured by the available leak detectors (measurands) — flow rate and concentration. We also need to know how to convert such quantities to get comparable results and understand the real implications of a leak.

The starting point for establishing maximum allowable leaks should be current requirements for natural gas. However, as hydrogen presents stronger flammable properties (larger flammability domain and lower ignition energy for instance) when compared to natural gas, we may expect some stricter requirements for the hydrogen gas and hydrogen-enriched natural gas network. Maximum allowable leaks are also crucial to define the smallest quantity that leak detectors should be able to measure or detect.

The main motivation for the detection of small leaks of hydrogen is to meet health & safety requirements. Hydrogen has a lower environmental impact when compared to methane, nevertheless, its global warming impact is not negligible when compared with CO_2. Only massive leaks can be economically relevant, but these leaks can be detected in real time by pressure drop or multiple flow measurements in pipelines. Thus, the key reason is the hazards of hydrogen - its potential easy ignition, an often invisible flame (but less radiative than methane flame), and its eagerness to burn or form explosive mixtures with air.

This report begins with a brief review of proper techniques for leak testing and leak location, and then discusses the different quantities measured by common hydrogen leak detectors. Then, it demonstrates the relationship between concentration and leak rate and summarizes common acceptance criteria required in different applications (scenarios). Finally, it discusses the use of reference leaks and how all measurements made by hydrogen leak detectors can be made traceable to reference quantities. At the end, the new requirements for the metrology infrastructure in Europe are also discussed.

2. Leak testing techniques

It is important to keep in mind that not all leak testing techniques require leak detectors. An overview of many techniques is provided by EN 1779 where 16 techniques different are compared. Technique selection depends on:

- Required tightness (EN 1779 covers the range from 10-8 to 10-2 atm.cc/s).

- Application (closed or open volumes, vacuum or under pressure, flexible or rigid walls…).

- Need of leak location.

- Need of quantification.

These techniques can be categorized in:

- Tracer gas detection.

- Pressure change.

- Bubble test.

While most of these techniques can be traceable to SI units, some are not. The latter is the case for the bubble test and all the techniques included in group B.1 (EN1779). These techniques detect a leak by a change in the colour of a coating or a tape, in the presence of the target gas, at the precise location of the leak. However, the detection limit of all techniques is defined. Other techniques such as ultrasonic and thermal detection, which are also difficult to trace to SI standards, are not included in the cited standard.

In the context of this project, the relevant techniques are those based on tracer gas detection using quantitative and selective gas detectors. Most of these detectors have been reviewed in a previous report in the frame of this project. However, hydrogen is not added in the gas grid to assist the detection of leaks but to replace natural gas. Nevertheless, detection of traces of hydrogen above its concentration in the atmosphere reveal the presence of leaks.

In certain fields, leak testing is part of the non-destructive testing group and must be carried out by well-trained personnel. ISO 9712 describes 3 levels of certification for technical personnel that are required by certain standards, codes or specific product specifications to supervise, interpret or perform leak tests.

3. Quantities measured by hydrogen leak detectors

There is a wide variety of leak detectors, measuring different quantities and covering very different ranges. The most common leak detectors measure the concentration of the target gas. Some can detect low ppm concentrations (volume/volume), while others work in the percentage range (≈ 0.1 to 100%). Some are portable, while others are stationary and suitable as room monitors.

Portable concentration detectors are useful for locating the leak. However, their reading depends on how close the probe is to the leak source, the angle between the probe and the flow direction, and the leak geometry. Hence having standardised sampling procedures is very important.

The concentration of hydrogen in the atmosphere is about 0.6 ppm. Therefore, this quantity can be viewed as the lowest useful concentration that leak detectors can measure. Even with background subtraction techniques, it is difficult to accept that concentrations below this limit can have any meaning in leak testing.

Another group of leak detectors has a small pump to suck the gas into the sensor. This type of detector is capable of measuring the actual leak rate if the probe is close enough to the leak source. Knowing

the pumping speed, these detectors can provide both the leak rate and the concentration. The relationship between the concentration c_{H2} and leak rate Q_{H2} is given by:

$$c_{H2} = \frac{Q_{H2}}{p \times S}$$

Eq.1

where p is the total pressure (1 atm) and S is the pumping speed of the probe. Very often $S = 1$ cc/s (cc = cm³) which means that if Q_{H2} is measured in atm.cc/s (or scc/s, scc = cm³ at standard temperature and pressure) then Q_{H2} and c_{H2} has the same numeric value. For example, a leak of 10^{-4} scc/s corresponds to a concentration of 100 ppm (10^{-4}). $S = 1$ cc/s is typical in mass spectrometry leak detectors.

Since the use of a pump dilutes the gas released by a leak (except when the leak rate is equal to the pumping speed), detectors without pump may be more sensitive than those using the same sensor attached to a pump. An exception must be made for leak detectors based on mass spectrometers, where the exceptional sensitivity, linearity and selectivity allow their use even well below 1 ppm (in controlled atmospheres). The main disadvantages are that these detectors have limited portability and a higher cost.

In addition to the different quantities measured by hydrogen detectors, some of them do not provide a numerical reading of the quantity measured, only a visual indication on a scale of LEDs with or without audible sound. Thus, it is not easy to establish a quantitative relationship between a reference quantity and the detector reading as required in a calibration. However, these detectors can be tested for their sensitivity to a traceable reference leak. This procedure is usually done for refrigerant gas leak detectors.

4. Converting concentration in leak rate

EN 13185 (under technique B.6) describes how the leak rate can be ascertained from concentration measurements if the leakage point is properly covered with a hood or bag. If this bag has a known volume V, then the leak rate is given by:

$$Q_{H2} = \frac{c_{H2}V}{t}$$

Eq.2

where t is the accumulation time.

However, the bag is often flexible and the volume in unknown. In this case, the use of a calibrated reference leak is very useful (leak rate = Q_{ref}). The leak is connected to the bag after the first concentration measurement Δc_1, at time Δt_1, without venting. The reference leak is left delivering the tracer gas for a controlled time Δt_2, and the increase in concentration is measured (Δc_2). From both, concentration and time measurements, a fully quantitative leak rate is obtained from the following equation:

$$Q_{leak} = Q_{ref} \left(\frac{\Delta t_1 \Delta c_1}{\Delta t_1 \Delta c_2 - \Delta t_2 \Delta c_1} \right)$$

Eq.3

In Fig. 1 this process is graphically illustrated, and the variables of the above formula are shown. For the data in this figure, the unknown leak comes 3.3×10^{-6} scc/s if the reference leak has a flow rate of 1×10^{-5} scc/s. If the detector resolution is known (or the minimum detectable change in concentration), then we can estimate the detection limit for reasonable accumulation times.
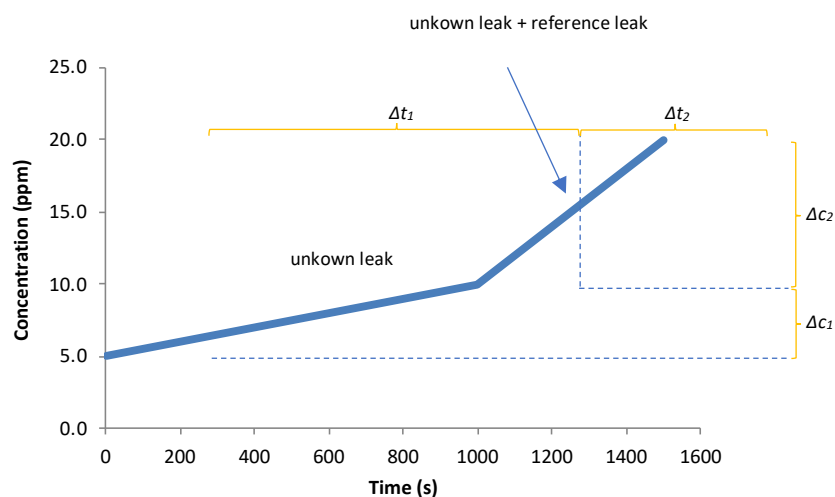
Figure 1- Concentration evolution in a bag (hood) produced by an unknown leak and by a reference leak.

EN 15446 also proposes an approach to convert leaks measured with concentration detectors in mass flow units. It is based in experimental data obtained in leak repair campaigns performed by the chemical industry in United States with various detectors. A set of correlation parameters (the so called SOCMI correlations) are provided that allow an estimate of the leak rate for volatile organic compounds (not applicable for H2). This standard requires detectors with a pump having a flow rate between ≈ 4 and 20 scc/s. As explained before, detectors with pumps can directly provide both, the leak rate and the concentration if the manufacturer includes this feature and if the sniffer is placed near to the leak point. The main need of converting concentration in leak rate are in detectors where the pump is not included.

A special reference should be given to the Bacharach Hi Flow Sampler which is an instrument specially designed to quantify methane leaks from industrial sources. This detector measures a range of emission rates by employing two sensor modes allowing concentrations from 0% to 100% to be measured. Leaks are encapsulated and pumped into the detector. The leak rate is provided by combining flow and concentration measurements. A similar instrument for hydrogen is so far unknow.

5. Concentration generated by a leak in an open space

The quantity that properly describes a leak is its flow rate in mass flow units, not the concentration generated by the leak in the surrounding space. The concentration depends on the elapsed leaking time, the instrument's proximity to the source of the leak and on local wind and on other variables that affect the dispersion of the gas. In an open space these variables are very difficult to predict. Even when the detector probe is in front of the leak, the measured concentration can vary considerably with the exact position and angle of the probe. In the case of detectors with pumps, the gas leak is forced into the probe, providing a more reliable assessment of the leak in flow rate units.

However, measuring the concentration in the vicinity of a leak is very useful for different reasons. First, it provides an immediate indication of the danger produced by such a leak. It tells how close the concentration is to the lower explosive limit (LEL) and can help to assess the corresponding explosive volume and mass, allowing orienting what actions to take quickly. Second, many leaks cannot be located exactly – the pipe may be buried or inaccessible. In this case, the actual leak rate can never be measured. However, by measuring the concentration, the extent of the leak is assessed, and remediation can be planned, e.g., replacement of suspect parts, no matter where the leak is precisely.

It is useful to discuss the relationship between the measured concentration at a given position and the leak rate that generates the measured concentration. The simplest model is based on Fick's law of diffusion. The radial flux Q driven by diffusion in a sphere (from its centre) is given by:

$$Q = (c_a - c_b)4\pi D \frac{ab}{b - a} \qquad \text{Eq.4}$$

where c_a is the concentration at distance a and c_b is at distance b and D is the diffusion coefficient. If we assume that at large distances (b) the concentration ≈ 0 then we can rewrite the equation before as,

$$c(r) = \frac{Q_L}{4\pi Da}\left[\frac{(b - a)}{b} - \frac{(r - a)}{r}\right] \qquad \text{Eq.5}$$

where Q_L is now the leak flow rate and a is any distance slightly larger than the leak diameter. The above equation is valid for $a < r < b$ and $c(b) = 0$.
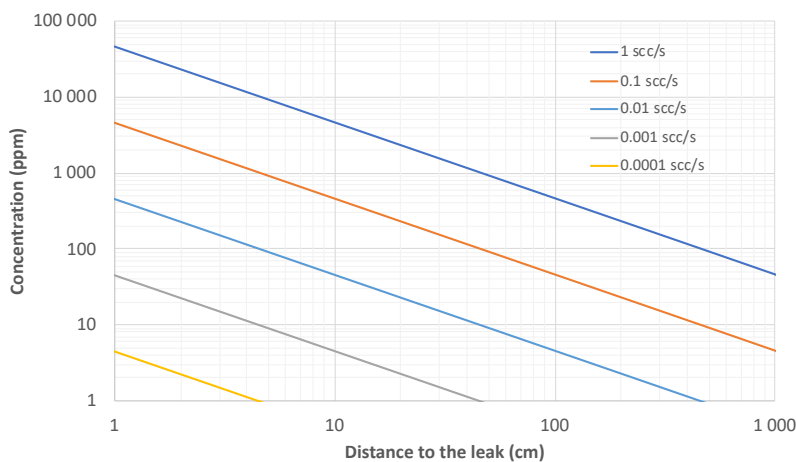
Figure 2- Relation between concentration and leak rate given by a simple diffusive model

Fig. 2 plots the result of eq.5 for different leak rates of hydrogen using a boundary condition of 0 ppm at b = 100 m. Although this is a very simple model, it does show the order of magnitude we can expect for the concentration produced by different leaks in an open space without any extra dispersion agents. We can see that the concentration increases directly with the leakage rate and decreases by 10 times when the leakage distance increases by 10 times. Only large leaks can be detected far from the source. The more sensitive the leak detector, the further away the leak can be detected. A detector with a detection limit of 1 ppm can detect leaks of 10^{-3} scc/s if the probe is at distances $\leq$ 40 cm. Detection capability for less sensitive detectors is evidently worse.

6. Time to achieve the lower explosive limit (LEL) in an enclosed space

One advantage of knowing the flow rate of a leak is that we can foresee when the lower explosive limit can be reached in a confined space without ventilation and considering a homogeneous distribution of the concentration. Single measurements of concentration do not allow for this prediction.

A leak with a flow rate Q leaking into an enclosed space with a volume V will generate a concentration equal to LEL in the time,

$$t_{LEL} = \frac{LEL \times V}{Q} \qquad \text{Eq.6}$$

Taking LEL for hydrogen as 4%, Fig. 3 plots the required time as function of the leak rate for different closed volumes. The orange dot in the graph shows the case of a leak of 10^{-3} scc/s flowing into a volume of 1 m^3. In this case, the time to LEL is 463 days.
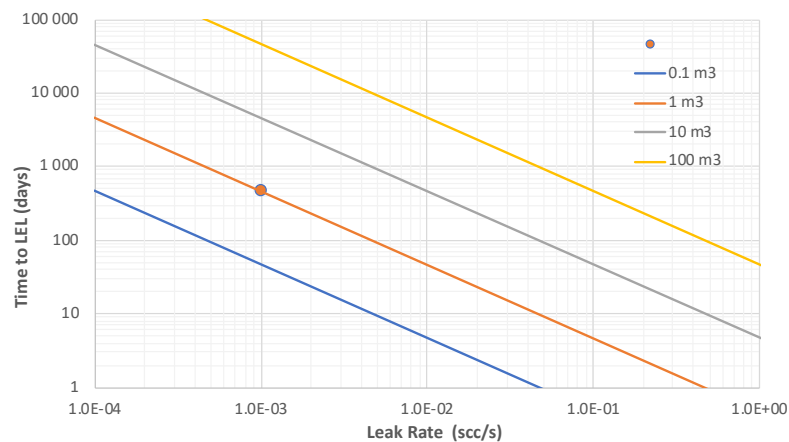

Figure 3- Time to achieve the lower explosive limit (LEL) of hydrogen as function of the leak rate for selected volumes considering a homogeneous concentration. The orange dot shows that time is 463 days for a leak of 10^{-3} scc/s in a closed volume of 1 m^3.

A volume of 1 m^3 is similar to the volume of some manholes in underground pipelines. In the extreme case of no ventilation and complete absence of hydrogen escape through the walls, the above formula provides a potential criterion for defining the maximum admissible leak and the frequency of leak detection campaigns.

Nevertheless, depending on the characteristics of the leak (flow rate and size) and on the size of the enclosure, the distribution of the concentration can be stratified. Thus, in this case, the LEL can be reached in the upper part of the enclosure, in a shorter time. The volume Richardson number is a criterion allowing predicting this phenomenon.

7. Leak testing scenarios and maximum admissible leaks

Maximum admissible leaks in hydrogen and hydrogen-enriched natural gas pipelines vary by application. Some parts are more critical than others. Also, pipelines in some areas have stricter requirements than in other areas. Some parts are factory tested; others are tested on site. Some tests are carried out during the commissioning phase, other tests are carried out during operation as part of a periodic maintenance and safety plan.

This section describes 3 possible scenarios and discusses some of the information found for maximum admissible natural gas leaks. This provides a starting point for proposing maximum admissible leaks once hydrogen is introduced in the gas grid.

Scenario 1 – parts manufacturing

In this section we may include items such as valves, pumps, compressors, gauges or even end-user domestic appliances. Most of these items are built in accordance with specific codes and have well defined requirements. Some international standards even describe the process and equipment to be used in leak testing.

For example, gas valves are tested to comply with ISO 15848 which defines as the tightest requirement a maximum leak of 50 ppm of He (as the tracer gas). This leak should be measured with a sniffer connected to a mass spectrometer, or similar instrument, with ≈ 1 scc/s pumping speed. Thus, 50 ppm will correspond to 5×10^{-5} scc/s. It is unknow if this requirement will change for hydrogen. Fuel pumps,

compressors and other critical parts have similar requirements and proper standards defining the acceptance criteria and testing procedures.

Burners and appliances burning gaseous fuels work with low gas pressures, just slightly above atmospheric pressure (≈20 mbar). Thus, tightness requirements are less strict. These appliances are typically tested with a pressure decay technique. Gas circuits are tested at a pressure several times higher than the working (relative) pressure (≥ 150 mbar). The acceptance criterion is in the order of 1×10^{-2} scc/s which correspond to ≈ 3×10^{-3} scc/s under operation conditions. This is well in the limit of bubble testing. Several standards provide clear criteria for these tests as EN 13611.

Scenario 2 – commissioning

Once a new section of the gas network is built, it is fully tested according to specifications and following well-defined procedures. At this time, it is possible to perform tests that are not possible to be performed in operation. Thus, we can expect that the tightness requirements at this stage can be more severe than when in operation.

Hydraulic tests are suitable for assessing mechanical strength but have limited sensitivity to gas leaks as the flow of liquids through small defects is limited by their surface tension and the hydrophobicity of the piping material. Suitable gas leak tests during commissioning are pressure drop, bubble test and accumulation technique in a hood with a tracer gas. The tracer gas is selected according to the leak detector. Common gases are 95%N2 + 5%H2 and helium.

The detection limit for pressure drop tests is limited by volume but extended by time. However, for a long time, temperature changes can mislead conclusions since thermal expansion or compression also changes pressure. A pressure drop of 1 mbar in a 1 m^3 pipeline after 1000 s corresponds to a leak rate of 1 scc/s, which makes this technique unsuitable for large pipelines. However, for pipelines in end-user homes, a smaller volume as 1 L provides a sensitivity of 1×10^{-3} scc/s. If the test is performed at a pressure higher than the working pressure, the time can be shortened while maintaining the same sensitivity. Contractors often use this test at the end of their work in low pressure applications. In the case of leaks found, the bubble test is used to locate the leak.

High pressure pipelines have stricter requirements, although quantitative data were not found in the literature. However, requirements such as those described for valves are expected to apply as well. Therefore, similar leak testing techniques should be chosen, providing quantitative flow measurements. Direct sniffing can be used, but the use of a hood (or bag) around the areas under test increases sensitivity and provides a way to test/pass large areas. The hood accumulates the tracer gas for a controlled time, say an hour or two. Then a reference leak is introduced to obtain a fully quantitative result (see eq. 3). In the presence of a leak, the probe is patiently swept over the suspected area to allow for its location. The detection limit of the accumulation technique is typically in the range of 1 to 10×10^{-6} scc/s but can be increased with long accumulation times and sensitive detectors.

Scenario 3 – under operation

Although the entire network is extensively tested prior to operation, many reasons can lead to subsequent changes in tightness leading to fugitive leaks. Therefore, many natural gas operators have regular LDAR (leak detection and repair) programs. These programs include surveillance with drones, cars and pedestrians equipped with gas detectors, but other approaches are also used, and advances are still being made in this field.

A reference document for this purpose is the "TECHNICAL RECOMMENDATIONS BASED ON BEST PRACTICES APPLIED BY EUROPEAN GAS SYSTEM OPERATORS" produced by Marcogaz and issued in April 2021. It describes the need for regular leak detection and quantification programs, as well as a list of techniques used. This document is based on a review of the procedures in use by European gas

suppliers. The only quantitative description of maximum allowable leakage is in its mention of the Dutch standard NEN 7244-6 applicable for distribution lines (p ≤ 16 bar). This standard categorizes leaks with 2 variables: (1) concentration produced by the leak and (2) distance from the source of the leak to the building. If the concentration is as low as 10 ppm and the distance is less than 0.5 m, the leak is in the "high priority" category and must be repaired within 24 hours. If the distance is 0.5 to 2.0 m, the leak is in the "low priority" category and must be repaired within 6 months. Fig. 4 shows these criteria graphically.
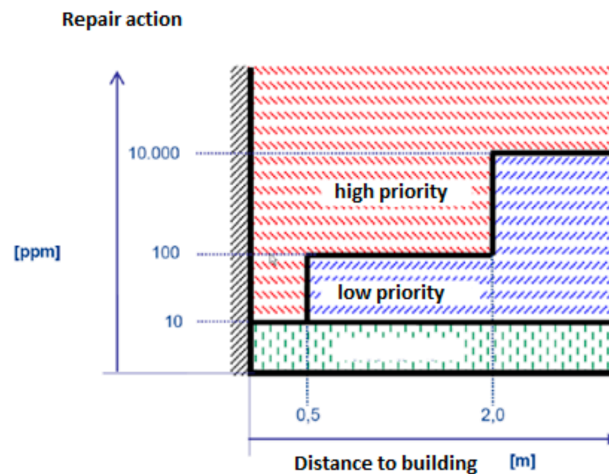

Figure 4- Reproduction of the criteria for leak survey from the Dutch standard NEN 7244-6

Marcogaz guidelines do not provide any quantitative information on maximum admissible leak rates. It states that emission rates can be derived from correlation factors as described in EN 15446, but correlation factors specific to the gas industry are required. Some gas operators, such as Enagás, have conducted internal studies to refine the correlation factors for methane. Quantifying leaks by measuring the methane concentration at the point of the leak and quantifying emission rates using correlation factors is currently an appropriate approach.

Other documents, while not defining maximum admissible leaks, do provide minimum requirements for the leak detector to be used. US ASME B31.8 (2003) states that the detector must be able to measure 50 ppm of gas in the air at any sampling point. The Dutch report KIWA GT-060241 in the section "Measuring Procedure for Above Ground Leakage Detection" requires measuring equipment to have a sensitivity such that a methane concentration of 10 ppm in air gives a meaningful indication. In the German DVGW G 465-4 standard it is written that the above ground test must be done with detectors with sensitivity ≤ 10 ppm. The British guide IGEM/TD/3 Issue 5 says that sensitive leak detection instruments, capable of detecting parts per million (ppm) of methane in the air, may be used to examine more closely all or parts of the system. In summary, leaks that generate concentrations of the order of 10 ppm are already significant.

8. Maximum admissible leaks for hydrogen

Defining maximum allowable leaks for hydrogen pipelines and hydrogen-enriched natural gas is not easy. First, leaks are measured in different quantities – concentration and leak rate. Second, different scenarios have different tightness requirements.

The main objective of this report is not to provide guidance to the industry, but to define how the metrology infrastructure should respond to future needs in this field to ensure traceability to SI standards of all measurements. However, the industry will certainly benefit from the above discussion

to select proper detectors and detection techniques and support the definition of maximum allowable leaks for each application.

Most of the earlier discussion was based on available documents for natural gas. Even for natural gas, maximum admissible leaks are not clearly defined, although maximum concentration levels (which trigger an action) have been found in certain documents. It is reasonable to expect that the increased risks resulting from the introduction of hydrogen into pipelines will result in an adaptation of these requirements, probably leading to an order of magnitude of less leakage.

Concentrations as low as 10 ppm are significant for natural gas. If the leak detector probe has a 1 scc/s pump, these concentrations correspond to a flow rate of 10^{-5} scc/s. Other conversion techniques should lead to leakage rates in the same order of magnitude. If the tighter requirements expected for hydrogen result in a lower factor of 10, it can be anticipated that concentrations as low as 1 ppm and leakage rates on the order of 10^{-6} scc/s could be significant. These should be lowest limit required of the metrology infrastructure.

9. Reference leaks

While reference leaks can be produced for a wide variety of gases and flow rates, it appears that such devices are not commonly used to quantify leak rates in the gaseous fuel industry. The benefits of these devices are discussed below.

Reference leaks are small portable artifacts that generate a predictable flow rate. Reference leaks in the range of 10^{-8} to 10^{-5} scc/s are normally produced with a gas reservoir and provide an almost constant flow. The depletion rate in this range is small enough to allow many years of near-constant flow without refilling the reservoir. Many of these reference leaks are produced with a permeation element (e.g., quartz or Teflon) through which the gas continuously permeates. Others have a very small open gas conductance. Many reference leaks with reservoir are produced with helium, but some manufacturers are able to produce leaks with other gases, including hydrogen.
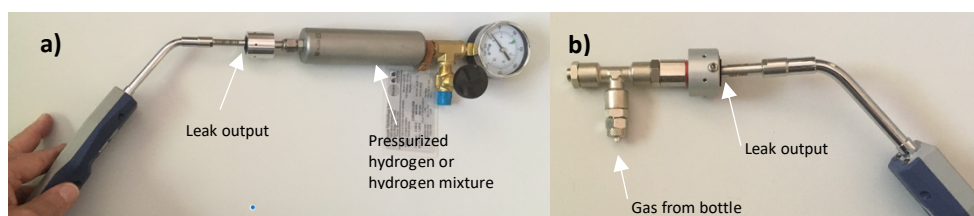

Figure 5- Sniffer probe sampling a reference leak; a) reference leak with reservoir; b) reference leak without reservoir, it should be connected to a bottle delivering a controlled pressure.

For ranges between 10^{-4} and 10^{-2} scc/s holes, capillary leaks or crimped capillaries are typically used. For higher flow rates, continuous microcalibrators, with a needle valve and flowmeter, are more convenient. Leaks within this range need to be connected to a gas reservoir with the target gas.

Reference leaks generate dynamic conditions, unlike calibrated gas mixtures which only allow static concentration tests. Thus, reference leaks simulate true leaks better than calibrated mixtures. Using reference leaks has the following additional advantages:

- Reference leaks can be calibrated in many accredited laboratories.

- Most references leaks are very stable and last many years without recharging.

- Reference leaks provide a prompt way to make the leakage measurement traceable.

- Reference leaks can be used to convert concentration in flow rate (or emission rate) following eq.3.

- The performance of leak detectors can be assessed at any time in the field by the technical personnel just by approaching the probe to the reference leak.

Another advantage is that a reference leak can be used to generate a concentration ramp if connected to a closed volume. For example, a reference leak of 10^{-5} scc/s connected to a volume of 1 L will generate a concentration of 1 ppm after 100 s, 10 ppm after 1000 s and so on. If the detector probe is connected to this volume, its linearity can be tested. If after a certain time the leak is diverted elsewhere via a 3-way valve, a static concentration is produced. The use of a fan inside will ensure a homogeneous concentration. This technique also has economic benefits, as this way of generating a traceable mixture is cheaper than using a certified mixture from a bottle of compressed gas.

Reference leaks are commonly used when leak testing is performed with helium by mass spectrometer leak detectors and in the HVAC & Refrigeration industry to verify the sensitivity of halogenated gas leak detectors.

A relevant detail is that reference leaks must be calibrated under the same conditions as for their future use. Many reference leaks are calibrated against vacuum, for internal use in mass spectrometer leak detectors. But for applications within the scope of this report, reference leaks must be calibrated against atmospheric pressure as is also the case of leaks for helium sniffers.

10. Calibration of leak detectors

Should leak detectors be calibrated in a laboratory? Like many other measurement sensitive devices, some leak detectors have limited time stability. Typically, the more sensitive the leak detector, the greater is the drift of the measured quantity. Therefore, many instruments are recommended to be calibrated in situ before use, not some time before in the laboratory. E.g., the accuracy of a leak detector in the range < 100 ppm will most probably drift significantly between calibration intervals. However, the use of a calibrated leak in situ will mitigate this problem since any measurement can be compared with that of the reference leak. Measurement traceability is immediately provided by the reference leak, no matter the detector is 'calibrated' or not.

Some leak detectors have an internal reference leak and perform a self-calibration each time they restart or whenever the user requests it. On all other sensitive leak detectors (< 1000 ppm), it is good practice to have a calibrated reference leak (or calibrated mixture) each time the leak detector is used. Higher range detectors (0.1 to 100%) are typically more stable, so they can be calibrated in advance.

For many applications, it is not important whether the leak detector reading is linear with concentration or not. It only matters if the detector is sensitive to the maximum allowable leakage for that application. Therefore, these detectors can be regularly tested in the laboratory for their sensitivity using a reference leak. In this case, there is no calibration, just testing against a well-defined traceable quantity.

In other cases, the detector is used only to provide a relative reading without the need for calibration. In the accumulation technique, whenever a reference leak is used, traceability comes directly from the reference leak. The detector only provides a reading which can be in any scale and unit. Any calibration factor in the amount Δc_n of eq. 3 will be cancelled. However, in this case, the output of the leak detector is expected to change linearly with the measured quantity.

Some general recommendations to ensure traceability of leak measurements are as follows:

- Leak detectors in the range of 0.1 to 100% should be calibrated in laboratory once a year.

- Sensitive leak detectors (< 1000 ppm) should be calibrated in situ with calibrated leaks or mixtures, unless its medium-term stability is well described by the manufacturer.

- All leak detectors may be tested in laboratory for their detection limit or alarm threshold with calibrated quantities. This includes all instruments without quantitative output.

- Calibration of reference leaks should be performed regularly, typically once a year.

- Detectors should never be tested with pure gases since the lifetime of some gas sensors is limited and sensors age faster with high concentration of gas (unless the manufacturer has different recommendation).

The use of reference leaks in field testing does not require a substantial extra effort. Every time a leak is found and require quantification, the reference leak is also measured. Calculations can be made ex situ to determine the true leak rate.

As a matter of example, most leak detectors used in the HVAC & Refrigeration industry are not calibrated but tested to confirm their sensitivity to a leakage of 5 g/year. ($\approx 4 \times 10^{-5}$ scc/s) as required by European regulations. Most of these detectors are quite sensitive but do not have a quantitative output, just a LED scale and a variable beep sound. These tests are performed annually at accredited testing laboratories with traceable reference leaks. The required uncertainty for these leaks is ± 15 % to comply with EN 14624.

11. Requirements for the metrology infrastructure

The traceability of hydrogen leak detectors should be done via materialized quantities as calibrated mixtures or reference leaks. If calibration is required at more than one point, multiple reference quantities should be used. Thus, European calibration laboratories should provide services for the calibration of:

- Mixtures of H_2 in N_2 (or in other suitable gas) in the range of 1 to 1000 ppm.

- Hydrogen reference leaks with flow rates in the range $[10^{-6}, 10^{-3}]$ scc/s.

Reference quantities within these ranges can later be used by others to calibrate or test leak detectors, in the laboratory or in the field. Different tests can be performed for hydrogen leak detectors such as selectivity, sensitivity, dynamic response, recovery time, linearity, etc.

The use of reference leaks has the advantage that it can also be used to generate static and dynamic mixtures of known concentration. However, mixtures produced by this process are less convenient whenever they are needed for immediate use, as is the case for testing concentration detectors in the field.

Regarding uncertainty, we must keep in mind that leak detection is not intended to provide an accurate measurement of the leak rate. The main concern is to guarantee that safety is ensured, and, in the event of a leak, appropriate measures are taken. Therefore, large safety factors are commonly introduced into acceptance criteria. However, adequate uncertainty is important for testing and comparing quantitative leak detectors. In this context, a maximum expanded uncertainty of 10% appears to be reasonable and technically possible for the lower limit of reference quantities.

The production of calibrated mixtures is available from many European NMIs (National Metrology Institutes) and from some gas suppliers. Although mixtures of hydrogen in inert gases are not readily available, most NMIs have well-defined procedures for producing such mixtures when demand begins. The lower 1 ppm is still within the capacity limit of many laboratories. Traceable calibration blends with 10 ppm hydrogen in nitrogen are offered by at least one commercial gas supplier in the US.

Calibration methods for reference leaks are well described in ISO 20486. This standard describes seven different calibration methods covering the full range and the two typical delivery pressures - vacuum and atmosphere. The most suitable primary method for hydrogen reference leaks is the G method, which is based on a constant pressure flowmeter. This method is suitable for working in the range $[10^{-6}, 10^{-4}]$ scc/s. In the range $\geq 10^{-4}$ scc/s other methods are also suitable such as method F. Both methods are suitable for any gas.

At the time of writing this report, the NMIs offering calibration for hydrogen leaks to the atmosphere are PTB in Germany and CMI in the Czech Republic. In addition, METROVAC which is an accredited laboratory in Portugal, also offers traceable calibrations of reference leaks of any gas. As many reference leaks have pressurized reservoirs, air transport has constraints. Therefore, it is important that more laboratories prepare to offer these calibration services as more hydrogen leak detectors are expected to start being used soon for leak detection in hydrogen and hydrogen-enriched natural gas pipelines.

In the frame of the present EMPIR project, validation facilities are being developed to test devices and methods of measuring hydrogen leakage by recreating real conditions in a testing rig. This work is being done at NPL and at Enagás and will allow to validate instruments, and more importantly, method performance. It will also assess the performance of individuals carrying out the measurements via Proficiency Testing schemes.

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