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A comparative study of odorants for gas escape detection of natural gas and hydrogen.

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Abstract

Decarbonising the residential heating and cooking sector is essential to meet national and international carbon emission reduction targets. Hydrogen has been identified by the scientific community, industry, and policy makers as part of the solution to this challenge. Hydrogen has been used for decades in many industries, formerly making up approximately 50% of the Town Gas used for heating and cooking in UK homes in the mid 20th century. It is now crucial to ensure safety regulations are met, and public acceptance gained, before hydrogen can start being used for residential heating. Demonstration projects require hydrogen distribution networks to be odorised. This study examines the use of sulphur-based odorants, which are currently in use in the UK and Europe to odorise Natural Gas, to be used in a 100% hydrogen gas demonstration network in the UK. We undertook a comparative testing programme to evaluate the escape detection properties of odorised hydrogen against odorised methane and natural gas. This comparative approach will help address the question asked by UK and EU regulators: is hydrogen ‘as safe as’ natural gas? The results show that untrained participants can identify an escaping gas odorised with Odorant New Blend and standby odorant 2, in hydrogen, natural gas or methane, at the regulatory threshold of 1% gas in air. These results contribute to the safety-case of H100 led by SGN.

Introduction

Hydrogen is currently being considered to support the decarbonisation of the energy systems which are, at present, overly reliant on GHG emitting natural gas. Ensuring that hydrogen is ‘as safe as’ natural gas is a critical requirement for this transition to happen (Kopasz, 2007). Odorising hydrogen to allow escape detection, without artificial sensors and training, is paramount in achieving the safety standards required for domestic use. Here we present the need for hydrogen in the current energy system and what it means

for innovative projects like H100 led by SGN for which this work was carried out. We then follow by an overview of the history of the odourisation of gases, and then highlight the remaining knowledge gaps that need addressing to ensure that the rate of hydrogen uptake allows decarbonisation targets to be met.

Hydrogen in a changing Energy System

Hydrogen gas has been identified as a significant contributor to achieving a low-carbon future. Hydrogen has been proposed as an alternative zero-carbon energy carrier, which can be converted to heat or electricity, with the potential to be easily stored and transported (Pudukudy, Yaakob, Mohammad, Narayanan, & Sopian, 2014). Hydrogen would also contribute to increasing the security of the energy system by reducing the dependency on fossil fuels (Sheffield & Sheffield, 2007). Most importantly it would act as a carrier allowing energy to be transmitted and used across the transport, heat, industry and power generation sectors (Staffell et al., 2019). Hydrogen could indeed allow the decarbonisation of the heat and transport sectors, which have proved challenging to decarbonise in comparison to electricity generation (Staffell et al., 2019). In the UK, for example, which relies on natural gas to heat 84% of its households, the gas emissions from the residential sector has reduced by only 16% from 1990 levels (BEIS, 2019). This illustrates that the rate of decarbonisation of heating needs to be increased rapidly (Staffell et al., 2019). Not only would hydrogen offer a way forward in speeding up the decarbonisation of the residential sector, but it would also offer end users the opportunity to keep using heat in a familiar and accepted way e.g. through combustion boilers and cookers, similar to the natural gas products in use today (Dodds & Demoullin, 2013). Hydrogen would also offer, with some network reinforcement, a decarbonised future to the vast national and international infrastructure used to deliver gas which has been upgraded over the past 50 years (Dodds & Demoullin, 2013).

Hydrogen 100 Project – Residential heating and cooking through a 100% hydrogen network

This work was commissioned by SGN and supports the safety case of the ‘Hydrogen 100’ innovation project. SGN is a UK gas distribution network operator. The Hydrogen 100 project seeks to demonstrate the feasibility of supplying 100% hydrogen to 300 selected homes which could include small businesses. The project will ensure that new 100% hydrogen compatible appliances are installed and a new hydrogen distribution network is built. We note that although a wider transition to hydrogen use would rely on both new and existing infrastructure, this demonstration project relies on new assets to reduce risk and uncertainty whilst gathering knowledge and experience. The work presented in this study investigates whether odourised hydrogen releases within a conceptual domestic space would be as readily detectable as a release from odourised natural gas. The Hydrogen 100 project includes a comprehensive investigation of hydrogen delivery through a new polyethylene distribution network. Further investigations related to odorants are being undertaken including physical testing of material exposed to odourised natural gas and hydrogen.

Brief history of gas odorisation

Mining has historically been an industry where exposure to the hazards of odourless combustible gases was frequent and deadly. When gas started to be used more readily in the 19th century in urban settings the idea of odorising this gas, before use, was proposed by Julius Quaglio (in 1880) in Germany (Fink, 2015). Although gas from natural sources often contains impurities providing a natural odour, processing techniques increased the odourless character of some pure gases like methane, carbon monoxide, carbon dioxide and hydrogen, making them more dangerous. Natural gas has been odorised for more than a century (Kilgallon, Gilfillan, Haszeldine, & McDermott, 2015). The odorisation of natural gas from distribution networks became a legal obligation following the ‘New London School’ accident in Texas (1937), which claimed the lives of 294 people (Kopasz, 2007). Odorisation is regulated because the primary component of natural gas, methane, is colourless and odourless. Since the same is true for hydrogen, artificial odorisation using an odorant compound would be required to meet regulations. Still to this day, in order to deliver gas to customers via a distribution network, gas needs to be odorised. Odorisation is a legal requirement in many countries because it is deemed an essential safety feature (Dodds & Demoullin, 2013; Puri, 2006). Indeed, most healthy people untrained in olfactory detection will be able to detect the distinctive smell associated with a gas escape due to the now widely enforced odorisation practices.

Research gap in hydrogen network odorisation assessment

Significant research has been undertaken on the odorisation of natural gas in pipelines. Some key points to consider with gas odorisation are the detectable limits of the gas odour, the type of odorant used (e.g. sulphur based, acrylate), the odorisation technique, and the monitoring system used to ensure that odorants meet the regulatory thresholds. These aspects are reviewed in Fant (1993).

The literature highlights both the importance of odorising hydrogen (Brewer, 1978) whilst acknowledging that the odorants might not behave in the same way in hydrogen as they do in natural gas. This is primarily due to the different chemical and physical properties of hydrogen (Dodds & Demoullin, 2013; Kopasz, 2007). The chemical compatibility of sulphur based odorants with hydrogen during piped transport has been demonstrated by the Health and Safety Executive of the UK (JP Hodges, Geary, Graham, Hooker, & Goff, 2015).

However, there remains a lack of reported investigation into the physical and olfactory properties of high purity hydrogen and odorant mixtures. This lack of investigation could hinder the progress of crucial demonstration projects needed to prove the use and deployment of hydrogen at scale. To date most of the focus on hydrogen odorisation has been placed on the end uses. In particular, fuel cells used to produce electricity from hydrogen (de Wild, Nyqvist, de Bruijn, & Stobbe, 2006; Dodds & Demoullin, 2013; Imamura, Akai, & Watanabe, 2005; Kopasz, 2007). This is understandable since commonly used sulphur based odorants are toxic to fuel cells, and fuel cells are likely to become a critical end use for

hydrogen (Staffell et al., 2019). Hence, a research focus to date has been placed on the desulfurization of odourised gas (Kim et al. 2007, Bae et al. 2009, Oshima et al. 2020). However, our study addresses the lack of investigation into the olfactory response to odourised hydrogen compared to natural gas, for an equivalent end use aimed at combustion appliances. This work complements a previous study by Mouli-Castillo et al. (2020) which investigates the likely olfactory characteristics of odourised hydrogen delivered to consumers via a distribution network. The Mouli-Castillo et al. (2020) investigation focused on applying standardised olfactory tests to odourised hydrogen. This method provided quantitative information, using reproducible tests, but not a direct comparative assessment with odourised natural gas. This comparative testing is important since the UK and EU regulators have sought demonstration (via comparative testing) that hydrogen is ‘as safe as’ the natural gas currently in use (HyDeploy website and (JRC, 2013)). In this study we report, for the first time, a set of comparative tests undertaken by Kiwa Gastec on odourised hydrogen, methane and natural gas from the UK network. We discuss the findings in the context of the wider Hydrogen 100 project, as well as the ‘as safe as’ approach sought by the regulators.

Odourisation Practices in the UK

In this section we briefly introduce the regulations and standards governing gas odourisation practices in the UK. In the UK, any gas supplied to end users from a network which has a pressure of 7 bar gauge or below should be odourised (Gas Safety (Management) Regulations, 1996).

The gas should be odourised to impart an odour with an intensity of 2 on the Sales scale (Sales, 1958), that is a ‘medium odour’. This will allow a gas escape to be detected by the population at 20% of the lower flammability limit, which is equivalent to about 1% gas in air concentration for both natural gas and hydrogen. In that sense odourisation is designed and regulated to provide warning prior to reaching flammable gas mixtures. Hence this study focuses on gas detectability rather than explosion risk, which is an important topic in its own right and is being actively investigated (e.g. Sinha et al., 2019; Lowesmith et al., 2009, Makarov et al., 2018). ISO 13734:2013 presents the general characteristics of an effective odorant:

- a) The gas odorant should have a strong odour at very low concentration,
- b) The odour character of the odorant needs to be unpleasant, distinctive and not confusable with other frequently occurring odours so that it is unmistakably associated with a gas escape,
- c) The odour character should be the same at different dilutions of natural gas with air,
- d) The odorant should be sufficiently stable during storage and when mixed with natural gas,
- e) The volatility of the odorant should be high enough so that the odorant does not appreciably condense under the conditions (temperature and pressure) existing in the pipeline system,
- f) Evaporation of the gas odorant should not appreciably leave residues,
- g) The odorant should be useable at low temperatures, when required,
- h) The combustion of the odorant should not leave significant solid deposits,
- i) The addition of the odorant to natural gas should not make the resulting gas harmful,

Currently demonstration projects, which are designed to underpin and support the uptake of hydrogen as a decarbonisation technology, are required to demonstrate that hydrogen can be used in a way which is 'as safe as' natural gas is today (HyDeploy, 2019). We have formalised this approach into two research hypotheses:

1. The odorants currently used with natural gas (generally sulphur based compounds) will be as effective when used with hydrogen.
2. Very small escapes of hydrogen are detectable in the same way as an escape of natural gas in a comparable room space.

These were tested through a series of comparative olfactory experiments that provided quantifiable, verifiable, and comparable data on the olfactory response of human participants to both 1) odorised hydrogen and 2) natural gas.

Methods

This section details the experimental setup, choice of participants, the rules the participants had to follow, and finally the experimental matrix presenting the tests performed.

Experimental Setup

The experimental setup was designed to allow control of as many parameters as possible to ensure reproducibility of the experimental conditions. The various elements of that setup are described below and visualised in Figure 1.

- 1) An enclosure made of timber and heavy-duty polythene sheets. The dimensions of the enclosure were 3 m wide by 6 m long and 3 m high, giving a total volume of 54 m³. These dimensions are representative of a ≈ 25 m² living room (with a floor to ceiling height of 2.5 m), which was typical for the 1970's when domestic housing was converted to natural gas, and was consistent with the standards of the time (LABCWarranty, 2019; Park, 2017). It is deemed appropriate to use this comparative basis, as it is still representative of the type of UK housing likely to use gas heating.
- 2) An extraction system was installed. It comprised two extraction hoods installed outside the enclosure above the doors, and an extraction point inside the enclosure. All extraction points were connected to an extraction duct leading outdoors. The system was design to clear gas from the enclosure, the door area, and the waste gas from gas chromatographs. This ensured that the space could be cleared from any gas prior to initiating each experiment i.e. resetting to baseline conditions.
- 3) A system to mix pure gas with an odorant rich gas mixture, and then inject it into the enclosure was designed. Injection in the range of 0.01 to 50 litres per minute could be used. The system

was designed to mix un-odorised gas with odorised gas to reach the 2 part per million volume (ppm) odorant concentration target (discussed below) in the injected gas stream.

- 4) A sampling system was put in place which allowed a continuous and controlled extraction rate to be set for each of the sampling points. This ensured a fresh sample was always available and that there was no lag between any of the sample lines. Ten sample lines were used in total – nine to sample the enclosure, and one to sample the air outside to provide a reference sample to ensure the accuracy of the equipment.

Figure 1: Experimental Setup. The height levels of the sampling points are as follow: Top at 2.7m, Mid at 1.5m and Low at 0.3m. The dimensions of the wall vents are 229 x 229 mm.

The experimental testing was conducted in a controlled, indoor environment in the Kiwa Gastec testing laboratories. This avoided the effects of wind on the dispersion of the gas and allowed rigorous control of the gas injection and dispersion, including for the final venting procedure at the end of each test. The tests were carried at ambient temperature, representative of dwellings in temperate climates, such as the UK. The results might not be representative of poorly insulated buildings in tropical, arid, and polar regions.

Sampling Procedure

Two gas chromatograph analysers (Inficon Micro GC Fusion) were used for measuring the levels of methane or hydrogen in the sampled gas in ppm. Each chromatograph used a molecular sieve-type column heated to 90 °C with an argon carrier gas injected at 69 Millibar (mbar). The chromatographs sampled each line in turn through the multipoint sampling valves and the gas in air concentrations (in ppm) was recorded. The analysis process consisted of a short purge with the sampled gas 15 s) followed by injection into the column within the chromatograph (100 ms) and then progression of the sample through the column (120 s). The multipoint sampling valves were moved to the next desired sampling line, and the process repeated. The concentration of the flammable gas in question was then calculated from the chromatograph, logged, and graphed in real time for visual verification. Using two

chromatographs in conjunction enabled for an offset logging sequence whereby the time delay between each sampling point being measured was reduced. The sampling order is presented in Supplementary Table 1.

Furthermore, the participants were not made aware of where in the enclosure the lines were sampling from. In addition, the participants did not know that the lines and sample points were fixed during the course of the experiment.

Tested Gases & Odorants

The gases used in this test programme prior to any mixing were:

- 1) Line gas from the local gas distribution network (odorised with odorant NB).
- 2) Un-odorised methane (reference fuel G20 with over 99% methane (EU Commission, 2017))
- 3) Un-odorised hydrogen (Grade 4.5, that is, with a purity of 99.995%)
- 4) Odorised methane and hydrogen (provided in bottles at 20 ppm odorant concentration)

Gases were supplied to the injection rig at a pressure of 0.5 bar. A target odorant concentration of 2 ppm was achieved by diluting the odorised gas (at 20 ppm) with the equivalent un-odorised gas according to the test specifications (Supplementary Table 3). 2 ppm is the concentration used in the UK gas network (Mouli-Castillo et al., 2020). The specification for the odorant and gas mixtures used for the experimental work were provided by the National Physical Laboratory, and supplemented their research programme on the odorisation of hydrogen for gas distribution networks.

Odorant New Blend (NB) is in use in the UK. It is used, along with its diluted form (known as Standby Odorant 2), by the UK distribution network operator SGN. Odorant THT is an odorant commonly used on the European mainland. Odorants using mixes of the compounds tested here are also used in Japan (Cagnon, 2011). Therefore, the findings from this study are directly applicable to support the development of hydrogen networks in many countries.

The odorant components used are presented in Table 1.

Table 1: Odorant composition used in the tested mixtures.

Odorant	Composition
New Blend (NB)	78% tert-Butylthiol, 22% Dimethyl Sulfide
Standby Odorant 2	34% Odorant NB, 64% Hexane
Odorant THT	100% TetraHydroThiophene

The injection rates used aimed for a nominal gas in air (GIA) concentration inside the enclosure of:

- 10,000 ppm (1%) which is the concentration of gas at the maximum level allowed by the regulator (about 20% of the lower flammability limit).
- 1,000 (0.1%) an order of magnitude lower than the regulatory threshold.
- and 500 ppm (0.05%) 20 times lower than the regulatory threshold.

This range of concentrations allows for reasonable assessment of gas escape detectability before dangerous levels of gas are reached, whilst avoiding 'false alarms' from very low concentrations.

Air Tightness

The air permeability of a 'new build' property was assumed a worst-case scenario (Crowther, Orr, Thomas, Stephens, & Summerfield, 2015). An air tightness test was conducted to ensure the experimental setup was representative of such worst case scenario. This test measured the enclosure's permeability to air. A new build property has an air tightness value of 10 m³/h/m² or less (Crowther et al., 2015).

An approved testing company undertook the testing at 0.5 mbar to EN 13829:2001. The 0.5 mbar pressure used for the air tightness test is dictated by the standard and not related to the injection pressure used in our tests. The air tightness test results (Supplementary Table 2) show that the test enclosure with open vents performed comparably to a new build property. All the olfactory testing described in this study was undertaken with the wall vents open to be representative of a new build property.

Test Programme

The test programme was divided into two phases. The first phase aimed to calibrate the equipment and understand the pattern of gas dispersion within the enclosure. These tests were completed using un-odorised methane and hydrogen, and enabled the determination of injection flow rates that would lead to the targeted gas in air concentration in the second phase. In addition, these tests served to test various sampling locations in the horizontal and vertical plane within the enclosure to ensure sampling locations were representative and were not biased towards any potential gas accumulations (e.g. in the corners of the enclosure). After each test, the enclosure and the space around it, were fully ventilated using the extraction system.

Phase 2 of the experimental programme involved tests using natural gas (line gas), odorised hydrogen, and odorised methane. At the start of each experiment, the enclosure was setup in the same manner. The wall vents remained open, the centre vent on the extraction system was shut, and an integrity check was performed on the gas injection and sample lines. The extraction vents from above either end of the enclosure were left open for the duration of the test to allow for the extraction of any escaping gas (including waste gas from the chromatographs).

Once the enclosure setup was completed, the test flow rates of both the odorised gas at 20 ppm, and the un-odorised gas, were set using the mixing rig (Supplementary Table 3). Once the flow rates had stabilised

to provide approximately 2ppm of odorant within the un-odorised gas the mixed gas was injected into the test enclosure via the open end of a 10 mm copper pipe. In order to simulate a domestic space in which no active mixing of the volume would occur, it was decided not to homogenise the gas mixture inside the enclosure. This implied that the actual gas concentrations inside the enclosure would vary (hence the nine sampling points). The reference value of the 'gas in air concentration' (GIA), which was used to set up the injected gas flow rate, was located at the centre of the enclosure (see curve labelled 'Mid B' in the results section).

During the gas injection phase of the test, 'sniff tests' were carried out at specified time intervals using the sampling ports (Supplementary Figure 1) which were connected to the ten sampling lines. Each sniff test lasted a few seconds before the participant left the test area. Each participant was a voluntary Kiwa staff member untrained in olfactory assessment. They were asked to smell each of the sample ports in turn and to record whether they could detect a smell. If a smell was detected the participant was asked to rate the intensity of the smell using the following scoring system of 1 to 5, otherwise a score of 0 was attributed. The gas was considered detectable when 50% or more of the trial participants detected a smell on a given sample line. The scoring system was based on a recognised odorant detection and perception scale; used for air monitoring and odour science (IAQM, 2018; Nimmermark, Schmidt, Jacobson, & Gay, 2005).

- 1) Very faint, may be identified as a gas escape
- 2) Faint but identifiable as a gas escape
- 3) Easily detectable and easily identified as a gas escape
- 4) Strong and easily identified as a gas escape
- 5) Very strong/unpleasant easily identified as a gas escape

Five minutes after the start of injection the first sniff test was performed. This was followed by one every 15 minutes during the injection period. The last sniff test was performed following the cessation of injection, once the concentrations had stabilised around the target level for that test. This final sniff test usually occurred 10 minutes after the stop of injection. Following the sniff tests, the participants were asked to complete a walkthrough of the test enclosure.

The walkthrough was designed as follows: each participant entered the enclosure through the door located at the opposite end to the injection point. They then walked (as a group) through the enclosure towards the injection point and exited the enclosure through the door located closest to the injection point (Figure 1). The walkthroughs lasted approximately 15 seconds each. Following the walkthrough, the participants were asked to comment on the strength of the odour inside the enclosure. The same scale as the sniff test was used. In addition, participants were asked to indicate where the first odour was detected. One of four locations could be indicated: 1) as soon as the door opened before entering the enclosure, 2) as soon as they entered the enclosure, 3) midway through the enclosure, 4) at the injection point.

The doors of the enclosure were kept closed until the end of the test (except to let the participant exit and enter the enclosure). As the focus of the study is placed on the detectability of gas escapes, the tests were completed after the end of the walkthrough and no assessment of the dissipation of the gas in the enclosure was conducted. Once the test had been completed the enclosure was vented to the outside using the central vent of the extraction system (Figure 1).

Trial Rules

This section describes the rules that the participants had to follow during the tests. Safety procedures are not described here in detail, only the rules pertaining to the scientific validity of the experiments. A complete risk assessment was however carried out to ensure the safety of the participants and the trial operators. Key safety measures which should be mentioned are that 1) the flammable gas/air mixtures within the test facilities were continually monitored and never exceeded 1% gas in air (GIA), 2) potential sources of ignition were not allowed in the enclosure, and 3) any potentially unaccounted for odorant risk was mitigated by rotating participants, exposing them to odourised gas for only seconds at a time, and asking them to vacate the test area immediately after each test.

The participants were not qualified in olfactometry and can be described as ‘naïve’ from that standpoint. It should be noted that olfactory literature indicates that increasing age tends to reduce olfactory sensitivity, whilst the effect of gender on olfactory sensitivity depends on several factors including the study, odorant compound tested, and the test task performed (Bliss, Schulz, SENGHER, & Kaye, 1996; James Evans, Cui, & Starr, 1995; Klimek, Gudziol, Owen, Pauli, & Hummel, 2000; Nguyen, Rumeau, Gallet, & Jankowski, 2016). To ensure any that age and gender factors were captured in the study, both female and male participants were selected over three age groups 1) under 30 years old, 2) 30 to 50 years old and 3) over 50 years old. For each test a mix of males, females and ages was ensured.

To evaluate the public’s response to an escape of odourised gas, standards cannot be used as they require olfactory experts rather than untrained members of the public (CEN, 2003). Using untrained participants presented a methodological risk since the participants might not have constituted a representative sample of the wider population. This could have introduced bias. To account for this bias, the research was designed as a comparative study, assuming that any bias from the untrained participants would apply equally to all gases and odorants tested. Further confidence can be had by comparing the findings with standardised tests’ results, although these usually lack the comparative element added by this study (see discussion section) (Mouli-Castillo et al., 2020). This allows a useful comparative analysis. Such comparative assessments are needed to develop quantitative risk assessments (QRAs) for demonstration projects.

To guarantee that no cross-contamination or ‘nose blindness’ occurred for the participants, the following procedures were enforced:

- 1) The location of each sample point within the enclosure was not communicated to the participant. This removed the opportunity for the participants to pre-assume a particular gas concentration, and therefore bias their judgment (e.g. hydrogen concentrations are higher closer to the ceiling, therefore odour should be greater too).
- 2) The participants did not know the gas injection rate and the target GIA (ppm) concentration. This avoided any expectations as to whether or not an odour 'should' be detectable.
- 3) For the entire duration of the test the participant were prevented from accessing the zone where the cylinders with the concentrated odorised gases (at 20 ppm) were stored. This avoided any exposure to the odorant via a gas escape from the regulator, or from the valve of the cylinder.
- 4) The participants were instructed to leave the test area immediately after each experiment (sniff test or walkthrough).
- 5) Participants were rotated regularly and limited to the number of tests which could be conducted in a day.
- 6) Between 3 and 4 participants took part in each test.

Experimental Matrix

The experimental matrix used for the phase 2 of the testing can be found in the Supplementary Table 3. A total of 18 tests were carried out. In test 1,2, and 3 it was observed that no odorant was detected at all. This was attributed to the odorant being retained by the pipework in the initial tests. This is common phenomenon and industrial pipes are commonly 'soaked' with odorants prior to being used for commercial purposes. For this reason test 1,2 and 3, were repeated as test 6,7 and 13 respectively.

Results

The following sections present the results of the Phase 2 tests (presented in Supplementary Table 3) for which odorant detection occurred. We also explicitly mention for which tests no detection occurred. For each test the following items are presented: the GIA concentration obtained during the test, the time at which the first detection via a sniff test occurred, and the time and gas concentration in the enclosure when the walkthrough was conducted. The results are presented in subsection for each gas-odorant pair tested. Each subsection presents the results for the different concentrations tested.

Natural Gas

The presence of natural gas (line gas) was detected during the 10,000 ppm GIA test 4 (Figure 2.a), but not during the two 1,000 ppm GIA tests 3 and 13.

Figure 2: a) Test 4, natural gas (line gas) at a target concentration of 1% GIA. b) Test 4 - Average intensity recorded for the sniff tests.

Following initial detection after 1h05min of the natural gas odour on the sample lines 1 and 4, the odour remained detectable throughout the rest of the test period (Figure 2.b). The greatest odour intensity was usually recorded for sample lines 1 and 2 (the top and middle sample points furthest from the injection point), and line 4 at the high level in the middle of the enclosure (Figure 2.b).

Hydrogen with Odorant NB

Hydrogen was tested with Odorant NB (the principal odorant used in the UK natural gas network).

During the 500 ppm, 1,000 ppm and 10,000 ppm GIA tests, gas odour was identified.

During the 500 ppm experiment, sniff detection scores were variable with a highest intensity of 1. This rose to a maximum of 2 during walkthrough (Table 2).

During the 1,000 ppm and 10,000 ppm experiments (Figure 3 and 5 respectively), once odours were detected in the sniff tests, the odour remained apparent for the remainder of the trial period. The maximum intensity during the 1,000 ppm sniff test was 3, with an average of 1 (Figure 3.b). This rose to 4 during the walkthrough of the enclosure (Table 2). The gas smell was first identified in the enclosure at an average GIA concentration of about 600 ppm (maximum GIA of about 1,400 ppm Figure 3.a). A smell was detected on all sample lines, with lines 1 and 2 (top and mid-level furthest from the injection point) perceived to have the highest intensity of smell.

Figure 3: a) Test 7, hydrogen and odorant NB at a target concentration of 0.1% GIA. b) Test 7 Average intensity recorded for the sniff tests.

During the 10,000 ppm experiment, 20 minutes after the start of injection, an odour with an intensity of 2 was detected (Figure 4.b). At which point the maximum GIA concentration within the enclosure was ~3,000 ppm, with an average of ~1,000 ppm (Figure 4.a). During the sniff tests the average odour level was 2 (Figure 4.b). This was lower than the recorded value during the walkthrough of the enclosure where an intensity of 4 was recorded by all participants (Table 2). Once again, the strongest intensity of smell was recorded on sample lines 1 and 2, followed closely by line 4 (top position in the centre of the enclosure).

Figure 4: a) Test 5, hydrogen and odorant NB at a target concentration of 1% GIA. b) Test 5 Average intensity recorded for the sniff tests.

Hydrogen with diluted Odorant NB (standby 2)

Hydrogen was tested with hexane-diluted Odorant NB i.e. standby odorant 2, which is used by SGN in the UK.

Once tests 5 to 8 had been performed it was determined that due to the intensity of the odorant, and the similarity between the standby odorant 2 and Odorant NB, if standby odorant 2 was detected at low GIA

concentrations, then testing at higher concentrations was not required, since Odorant NB had been detectable at higher concentrations.

The presence of gas was not identifiable with standby odorant 2 at 500 ppm GIA concentrations.

During both the 1,000 ppm sniff tests and enclosure walkthrough, hydrogen odorized with standby odorant 2 was reported at a maximum intensity of 3, although the average intensity oscillated between 0 and 1 (Figure 5.b). Some of the participants identified it as a gas escape in their comments. At the time of first detection during the sniff tests, a maximum GIA concentration inside the enclosure of around 1,600 ppm was recorded, with an average GIA inside the room of about 650 ppm (Figure 5.a). Once identified the gas odour stayed evident during the remainder of the sniff tests.

Once again, the strongest intensity of smell was consistently recorded on sample line 1. Following the initial detection, the odour was identified on all the sample lines as presented in Figure 5.b.

Figure 5: a) Test 10, hydrogen and standby odorant 2 at a target concentration of 0.1% GIA. b) Test 10 Average intensity recorded for the sniff tests.

Hydrogen with THT

During the test of hydrogen odorised with odorant THT, gas was identified by the participants during the 10,000 ppm GIA concentration test (Figure 6.a). However, it was not detected for the tests with a GIA concentration of 500 ppm or 1,000 ppm.

The sniff tests conducted during the 10,000 ppm GIA concentration experiment revealed that the maximum odour intensity, which could be identified by the participants, was 2 (Figure 6.b). The participants also recorded that the smell was unpleasant but not immediately identifiable as a gas escape. The odour character was described as “chemically” or “similar to onion/garlic”. At the time of first detection, 1h05, by sniff tests the maximum GIA concentration within the enclosure was about 8,500 ppm, with an average of around 5,400 ppm (Figure 6.a). As in most of the previous tests, the highest odour intensity levels were recorded on sample lines 1, 2 and 4 (Figure 6.b).

The odour intensity registered by all but one of the participants during the walkthrough was of 4 (Table 2). This is higher by two scale points than during the sniff tests. A participant commented that the enclosure had a musty odour, whilst only scoring that odour at an intensity score of 1. The participants characterised the odour as “chemically” or “very strong onion” or “garlic”.

Figure 6: a) Test 18, hydrogen and THT at a target concentration of 1% GIA. b) Test 18 Average intensity recorded for the sniff tests.

Methane with NB

Methane (G20) combined with Odorant NB at 2 ppm was tested. The presence of gas could be identified by smell by the participants during the 10,000 ppm GIA concentration test (Figure 7.a). No odour was detected for the tests conducted at GIA concentrations of 500 ppm and 1,000 ppm (tests 15 and 16).

The maximum odour intensity recorded in the sniff tests was a value of 4 (Figure 7.b). The detection occurred after 20 minutes of injection (Figure 7.b). The maximum odour intensity of 2 at the time of detection during the 10,000 ppm experiment (Figure 7.b). At the point of first detection, the highest GIA concentration within the enclosure was approximately 2,000 ppm, with an average of about 700 ppm (Figure 7.a). The average odour intensity as indicated on each sample line by the participants is shown in Figure 7.b.

During the walkthrough a maximum odour intensity of 4 was recorded inside the enclosure (Table 2).

This intensity is consistent with the maximum identified during the sniff tests.

Figure 7: a) Test 17, methane (G20) and odorant NB at a target concentration of 1% GIA. b) Test 17 Average intensity recorded for the sniff tests.

Odorant Tracking

Table 2 displays the walkthrough findings during which gas was identified and ranked on odour intensity. It indicates at which location the scent was identified—"Door Open" means the odour was perceived as the door opened before the participant entered the enclosure.

The entrance door was furthest from the point of injection. The point of injection was located at the end of the enclosure. Odour identification took place at the 'entry to enclosure' (Table 2). This is evidence that the odorant persisted within the gas as it passed through the room, or that the concentration of odorant within the gas was sufficiently intense to be detectable everywhere within the release area.

The detection of the various odorised gases during the 'sniff tests' at sample lines 1, 2 and 3 (located furthest and closest to the injection point) are consistent with the observation that odorants do indeed remain within the hydrogen. It is conceivable that after the odour was identified the participants became used to the odour, and thus the lines which were sampled last appeared less intense. In some cases, the participants of the experiment were asked to reverse the order in which they checked the sample lines (still without knowledge of which sample line corresponded to which sample point), however this led to only minor variation in the apparent strength of the odour.

Table 2: Odour intensity recorded by participants during the walkthrough of the enclosure at given target locations. The letter does not identify a specific participant.

Test	Participant	Door Open	Entry to enclosure	Middle of enclosure	Point of gas injection
4 - 10,000 ppm Line Gas	A	0	0	4	4
	B	0	0	3	3
	C	4	4	5	5
	D	4	4	5	5
5 - 10,000 ppm Hydrogen NB	A	4	4	4	4

	B	4	4	4	4
	C	4	4	4	4
	D	4	4	4	4
7 - 1,000 ppm Hydrogen NB	A	0	3	3	3
	B	0	3	3	3
	C	0	3	3	3
	D	0	3	3	3
8 - 500 ppm Hydrogen NB	A	0	2	2	2
	B	0	2	2	2
	C	0	1	1	1
	D	0	2	2	2
10 - 1,000 Hydrogen Standby 2	A	3	3	0	3
	B	2	2	0	0
	C	2	2	1	1
12 - 1,000 Hydrogen THT	A	2	2	1	0
	B	0	0	0	0
	C	0	0	0	0
	D	2	2	1	1
17 - 10,000 Methane NB	A	5	5	5	5
	B	4	4	4	4
	C	0	4	4	4
18 - 10,000 Hydrogen THT	A	4	4	4	4
	B	4	4	4	4
	C	1	1	1	1

Discussion

This study tested the two hypothesis that 1) the odorants currently used with natural gas will have a similar effectiveness when used with hydrogen, and 2) that the detection of very small escapes of hydrogen are detectable in a similar way to a natural gas escape in an equivalent room volume. This section discusses how the findings address these hypotheses and outlines the results limitations.

The study was designed around the UK's legal requirement that the presence of gas should be readily detectable by smell at GIA concentration of 20% of the lower flammability limit. This equates to around 1% GIA for both methane and hydrogen (Gov, 1996). At this 1% GIA concentration, all the tested odorants led to the gas releases being detected by the trial participants. When mixed with either hydrogen or methane, both Odorant NB or Standby odorant 2 led to the smell being identified specifically as a gas escape at concentrations of 1% GIA. As such, it can be concluded that, under the conditions of this trial, the two hypothesis are verified.

During the hydrogen THT tests the odorant was detected at 1% GIA concentration, but not identified specifically as a gas escape by participants. The trial participants being UK residents could explain why they did not identify a gas odorised with an odorant primarily used on the European mainland. This result

suggests how embedded the odour character of gas is in a given population and corroborates the observation made by a report for the Health and Safety Executive of the UK (J. Hodges, Geary, Graham, Hooker, & Goff, 2015). This illustrates that, should a different odorant to the ones being used today be used in future hydrogen networks, the public should be correctly informed and educated to associate the new odour character to a gas escape.

Another observation of note is that odorant NB, currently in use in the UK, was detected at much lower concentrations (down to 0.05% GIA) when mixed with hydrogen in comparison with a detection occurring at 1% GIA when mixed with methane, or as part of line gas. However, the testing method does not allow for the cause of this to be determined with certainty. In addition, the results indicated that stratification of the gases within an enclosure 3 m high does not negatively affect the detection potential of a gas escape.

Additionally, we note that the THT target concentration in the gas was set to 2 ppm as per odorant NB and standby odorant 2 in the UK. However, literature suggests that the THT concentration in use in the European mainland are in the range of 3 to 11 ppm (Uni. Of. Miskolc & Hungarian Scientific Society of energy Economics, 2008; Zhang, 2019). This could explain why THT appeared to be less easily detectable than odorant NB.

Table 3 summarises that, for odorant NB and Standby odorant 2, the maximum and average GIA concentrations within the enclosure are comparable between the hydrogen and methane tests. This also confirms the hypotheses tested.

One important experimental design choice to consider is the use of volumetrically equivalent gas injection rates, as opposed to energy injection rates. Indeed, due to the lower energy density of hydrogen at the standard delivery pressure of domestic gas networks, the volumetric flow rate of hydrogen would be about 3 times greater than that of natural gas to ensure an equivalent rate of energy delivery. However, what our results have shown is that both hydrogen and methane escapes can be detected at equivalent GIA concentrations and both at the required value of 1% GIA.

Table 3: Maximum and average gas in air concentrations within the enclosure at the point of first detection.

Gas in air saturation (ppm)	Test 4 Line Gas 10000 ppm	Test 17 Methane + NB 10000 ppm	Test 5 Hydrogen + NB 10000 ppm	Test 7 Hydrogen + NB 1000 ppm	Test 10 Hydrogen + Standby2 1000ppm	Test 18 Hydrogen + THT 10000 ppm
9000 - 10000						
8000 - 9000						
7000 - 8000						
6000 - 7000						
5000 - 6000						
4000 - 5000						
3000 - 4000						
2000 - 3000						
1000 - 2000						
0 - 1000

Maximum ppm in the enclosure at the time of first detection

Average ppm in the enclosure at the time of first detection

This study found that odorants appeared to remain with the hydrogen gas as it moves through an enclosed space. Sprague and Flynn (2013) discussed that the molecular weight and dispersion properties of hydrogen, relative to odorant compounds, were likely to lead to the odorant not remaining within the gas stream in a stagnant environment (US 8 394 553 B2, 2013). However, they also suggested that in a domestic dwelling, ventilation could be sufficient to drive dispersion and to keep the odorant mixed with the gas stream (US 8 394 553 B2, 2013). The finding of this study supports this for hydrogen, natural gas and methane.

One limitation of the study is the small number of participants in each test (3 to 4). The variation in participant responses is apparent in a few tests. Detection occurred at 2000 ppm for test 17, whilst gas concentration in tests 15 and 16 reached a maximum of 2500 ppm without the gas release being detected. A similar observation can be made for test 12 where the escape was detected (but not identified as a gas escape) at a lower concentration than the maximum measured in test 11, when no detection occurred in test 11. This can be explained by the definition of “detection” used in this study to be when at least one participant detected a smell. This increases the sensitivity of the results to the individual participant’s olfactory sensitivity. Despite these variations, if we compare our findings to previous olfactory testing from Mouli-Castillo et al. (2020), which was performed on odorised hydrogen using standardised tests in an accredited laboratory, we find that the odorant concentration in air from our work are within an order of magnitude from those presented in that study. More specifically, our detection thresholds for Hydrogen with New Blend, Standby Odorant 2 and THT, are 6.15, 4.5 and 2.2 times greater than the ones from that study respectively. This can be explained, in part, by the use of ‘naïve’ participants in our

study, compared to an expert panel in their study. Another contributing factor is likely to be the fact that our study mimic a real new dwelling environment with natural ventilation resulting in uneven concentrations in the test volume, whereas olfactory measurements undertaken in their study are carried out on precisely measured samples. It should also be noted that a variation of less than an order of magnitude between olfactory studies with different methodologies should be considered a good match (Murnane, Lehocky, & Owens, 2013). Both studies' conclusions agree that odorant in hydrogen will induce an equivalent olfactory response to odorant in natural gas.

Conclusion

This research is important because it addresses a key safety aspect of gas distribution: the odourisation of gas to allow untrained people to detect gas escapes before they reach a flammable level. This research is essential to the safety cases developed by hydrogen demonstration projects, in particular H100 in the UK which aims to deliver 100% hydrogen to 300 homes via a purpose built distribution network.

By providing a direct comparison with methane, an approach which was not directly studied in the standardised tests from Mouli-Castillo et al. (2020), it addresses the key requirement imposed by regulators in the UK that hydrogen distribution should be demonstrated to be 'as safe as' the current natural gas one.

Our work provides evidence that firstly, the odorants currently used within natural gas will have a similar effectiveness in allowing escape detections when used with hydrogen. Secondly, that small escapes of hydrogen are detectable in a comparable way to a natural gas escape in an equivalent room volume. These conclusions can be considered robust as they were demonstrated by two different methodologies using very different approaches.

These findings are also applicable to other innovation projects around the world which require the demonstration that hydrogen can be effectively odourised and any releases detected. In particular, since the odorant compounds tested are used in many countries.

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Author Contributions

The work was sponsored by AM and MW from SGN. The experimental programme was designed by MEC from Kiwa Gastec; experimental work and data analysis was carried out by GO, JT and NH. JMC performed complementary analysis and carried out the literature review. The manuscript was written by JMC and GO, with contributions from the others.

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Competing Interest Statement

We certify that any and all of our affiliations with, or financial involvement with, any organization or entity with a financial interest in or financial conflict with the subject matter or materials discussed in the manuscript are disclosed in the funding acknowledgments section of this manuscript.

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