

Proposing a new tracer gas for future field applications of passive tracer gas tests for air change rate measurement

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ABSTRACT

This paper describes the ongoing development of a new adaptation of the traditional tracer gas test (TGT) used for total air change rates (ACH) measurement. This adapted TGT, based on constant tracer injection, is intended for use in large-scale IAQ assessments and employs an alternative tracer gas that is more adequate than the currently employed SF₆ and perfluorocarbons, and that can be co-captured and co-analysed with commonly assessed VOCs by commercial passive IAQ-sampling. Via literature study and lab experiments, decane-D₂₂ was found to be a suitable tracer substance. A passive source of decane-D₂₂ was developed and optimized in lab, providing stable and repeatable emission rates under standard temperature, while unaffected by varying RH and ACH. The effect of the liquid solvent level over the source emission rate was only barely noticeable, but a range of adequate solvent level is suggested nevertheless. The selected tracer was also shown not to adhere/absorb significantly to surfaces. Additionally, a consistent exponential curve was derived for determining the source emission rate from the room temperature. Field applications of this new TGT adaptation are ongoing and will be published elsewhere shortly.

INTRODUCTION

Indoor air quality (IAQ) is a topic of great public concern. Most people tend to spend most of their time indoors, where numerous known sources of harmful pollutants are typically present. The association between poor IAQ and adverse health effects has been shown in numerous studies (Fernandes et al., 2009; Heinrich, 2011; Carrer et al., 2018). A key factor that influences the accumulation of pollutants in indoor spaces and the interpretation of IAQ data is ventilation. However, due to the additional cost and the complexity of aligning ventilation measurements with IAQ assessments, only few IAQ field studies report ventilation rates adequately, i.e. fully describing the measurement method (Persily, 2015). Given the importance of ventilation in understanding IAQ, estimating pollutant sources' impact and proposing remediation actions, it is crucial that IAQ assessments

report actual ventilation rate values, measured by reliable methods.

Most ventilation assessments use a tracer gas test (TGT) as a method to measure total air change rates (ACH) in indoor spaces (Persily, 2015). In a TGT, the air is marked by the injection of a tracer gas and the ACH is then calculated from the tracer's emission rate and final room concentration. The TGT is the only method capable of measuring the actual airflow between building zones and the outdoors (Lunden et al., 2012). Although the TGT approach may present a relatively higher degree of uncertainty compared to other methods, this is compensated by its greater simplicity, convenience and possibility to be executed during normal occupancy (Lunden et al., 2012). TGTs are especially suited for large-scale ventilation surveys, in which a lower degree of individual data precision is acceptable in favour of an increased amount of data.

However, current TGTs present three important shortcomings: 1) Most TGTs provide instantaneous results (due to the use of online monitors), while the concentrations of many indoor pollutants are commonly measured by means of long-term (usually passive) sampling techniques that report time-averaged values, i.e. IAQ and ventilation data are not directly comparable; 2) Considering the passive-techniques-based TGT options (i.e. TGTs which employ techniques based exclusively on diffusive processes, using no pumps or electricity), most current applications employ either sulphur hexafluoride (SF₆) or perfluorocarbons (PFTs) as tracer gases, both being potent greenhouse gases with very long lifetimes in the atmosphere (IPCC, 2007); 3) Many passive TGT applications assume that the indoor air is perfectly mixed across the assessed space (which is not always the case in real life, i.e. the characteristics of the indoor air, such as temperature and pollutants' concentrations, can vary spatially across the same space), meaning that the placement of tracer sources and samplers can influence the measurements of tracer concentration, and potentially lead to severe bias in the calculated ACH values (Van Buggenhout et al., 2009; Lunden et al., 2012; Liu et al., 2018).

Therefore, the main goal of the project described in this paper is to develop an adaptation to the traditional TGT technique so it tackles the three aforementioned

shortcomings by: 1) utilizing commercially-available standard passive samplers, commonly used for IAQ assessments, to capture the tracer gas simultaneously with other pollutants of interest, thus matching the timescales of the datasets and enhancing their comparability, 2) using an alternative substance as tracer gas, more adequate than the currently used SF₆ and PFTs and 3) including a pre-test planning phase in which the air mixing in the space to be assessed is evaluated beforehand by means of computer simulations, so that, if needed, the potential bias arising from imperfect mixing can be minimized via optimizing the physical placement of sources and samplers. Additional advantages of this adapted TGT are its suitability to use during normal occupancy, as it causes no disturbance and employs a harmless tracer at safe air concentrations, and the employment of purely passive techniques, which lowers the costs and broadens the range of buildings where the TGT can be applied.

This paper focuses specifically on the work executed to select an alternative substance for use as tracer gas and to develop a suitable emitting source design for this new tracer. The issues related to air mixing and planning of physical sources/samplers' placement are discussed elsewhere.

METHODS

As mentioned above, this paper focuses on the processes of selecting an alternative substance for use as tracer gas and of developing a suitable emitting source design for the selected tracer. Both processes are described in detail below.

Selection of an alternative substance for use as tracer gas

A comprehensive literature review was performed to propose an adequate substance to be used as a tracer in the new TGT, alternatively to the currently used SF₆ and PFTs. To help guide this process, 6 determining criteria for a suitable tracer gas were set:

1. The tracer gas should be quantifiable by means of a commonly used passive air sampling method;
2. The tracer gas should be able to be analysed together with common IAQ pollutants;
3. The tracer gas should have negligible presence in typical indoor environments, thus having no significant known indoor sources;
4. The tracer gas should present no significant health impact, thus being suitable for use indoors during normal occupancy (including of most vulnerable populations);
5. The substance selected as tracer gas must be financially adequate, considering the amount needed for running a complete test;
6. The tracer gas should not be susceptible to physical and chemical parameters of the indoor environment.

Criteria (1) and (2) are directly aimed at eliminating the issue of time-scale discrepancy between indoor pollutants measurements and ACHs measured by this new TGT. The approach proposed by these two criteria also saves time and resources, as only one sampler and one analysis simultaneously provide all the information needed to infer both the ACH and the IAQ level of the assessed space. Since volatile organic compounds (VOCs) are arguably the most relevant gaseous contaminants in IAQ studies, they were selected as the group of substances from which the alternative tracer was to be drawn. The VOCs considered for use as tracer were those capable of being captured by the passive VOC samplers commercialized by Radiello® (Radiello, 2007).

Initially, paraffins were considered as candidates for their high stability, inertness and low toxicity. However, paraffins do not fulfil criterion (3), as they commonly present considerably high background concentration in most indoor environments. Therefore, the use of paraffins as tracer gases was considered inadequate.

An effective way found to circumvent this issue was the use of stable isotope labelling, more specifically of deuterated compounds. These compounds are, by definition, not naturally present in the atmosphere or in any household product. Stable isotopes are analytically distinguishable yet chemically and functionally identical to their original correspondent compounds (Wilkinson, 2016). Thus, the substances considered as alternative tracer gas were the deuterated paraffins in the C8-C12 range. In this range, decane-D₂₂ was selected for its combination of lower flammability and higher volatility.

To ensure the fulfilment of criterion (4), advice from the Flemish Institute for Technological Research's (VITO's) Exposure Modelling and Risk Assessment (Environmental Risk and Health Unit, BREM-G) on safe exposure to decane-D₂₂ levels was sought. From their report, decane-D₂₂ safety information is considered as the same as for n-decane, for which there is no harmonized classification. DNELs (derived no effect level) values, threshold concentrations for toxicity below which exposure is safe, have not been derived as n-decane is not hazardous for quantifiable effects (EC, 2006). There is also no derived OEL (occupational exposure limit) for n-decane. Nevertheless, the final advice from VITO's Exposure Modelling and Risk Assessment is: decane-D₂₂ concentration must not exceed 250 mg m⁻³ during generation of a stable airborne concentration for 1 day. The peak (<15 min) concentrations should not exceed 500 mg m⁻³. Also, as the substance is flammable, there must be no open flames, no sparks and no smoking during a TGT using decane-D₂₂ as tracer. Above 46°C explosive vapor/air mixtures may be formed; thus, temperatures must be kept below this limit.

Regarding criterion (5), an ampule containing 5 g of decane-D₂₂ is sold by Sigma-Aldrich® for €491,00. Considering that the intended emission rate of the tracer source is in the magnitude of 1 mg h⁻¹, a 5 g ampule should provide enough tracer for more than 6 months of continuous emission. Therefore, for the purposes of the present paper, it is considered that decane-D₂₂ meets criterion 5 as well.

Criterion (6) is the only one that cannot possibly be evaluated via literature review. Ideally, the behaviour of the tracer gas, during and after injection to the studied space, should be absolutely independent from environmental conditions, such as temperature (T), relative humidity (RH) and ACH itself. Moreover, the tracer should not have any significant sinks in common indoor spaces (e.g. adherence and/or sorption to varied surfaces). However, by the own nature of the diffusion and sorption processes involved in passive emission and sampling, room conditions (especially temperature) can significantly affect the source's emission rate. Therefore, criterion (6) was evaluated in the form of a long series of lab tests involving replicate tracer sources and test chambers for controlling the environmental conditions.

Physical source designing and testing

In practice, the application of a TGT requires the placement of a source (or several sources) that injects the tracer gas in the air of the assessed space and a sampler (or several samplers) that monitors the concentration of the tracer gas. As explained above, the type of sampler to be employed in the new proposed TGT was pre-determined: commercial adsorptive samplers for passive long-term sampling. Given this project's focus on simplicity and inexpensiveness, it was decided that the employed source should also be passive and based on the constant emission approach (Persily and Levin, 2011). In this approach, the source emits tracer gas at a constant rate until the room concentration reaches a steady state (assuming a constant ACH), from which the total ACH can be inferred using Equation 1.

$$q = \frac{G}{V C_s} \quad (1)$$

where q is the ACH [h⁻¹], G is the source emission rate [μg h⁻¹], V is the total volume of the assessed space [m³] and C_s is the steady-state tracer concentration [μg m⁻³]. (If q is not constant, the final concentration measured by the passive sampler will not be C_s , but rather the average concentration over the sampling period. Thus, Equation 1 is still valid, but in that case the calculated q represents the average ACH over the total period).

Several options were tested in pursuit of the most adequate source design, i.e. one that allows a stable, repeatable and significant tracer emission rate using a small amount of liquid solvent. Figure 1 shows the different types of source design that were considered at the beginning of the testing process.

The two designs shown in Figure 1a were discarded early on because they would require a relatively high volume of liquid decane-D₂₂ compared to the other designs. The design shown in Figure 1b (design B) is based on the source used in a previous study by Shinohara et al. (2010), which consists of a 1 ml glass vial filled with the tracer in liquid state and placed inside a 5 ml glass vial, both capped with metal caps. The tracer emission is initiated by piercing a needle (0.4 mm diameter, 20 mm length) through both caps to reach the headspace of the smaller vial. The needle is then attached to a plastic syringe (9mm diameter and 7.5 cm length), which acts as the tracer diffusion path, where a polyethylene (PE) sintered filter disk is placed to help maintain the emission rate. This design (originally intended for PFT's emission) yielded too low decane-D₂₂ emission rates.

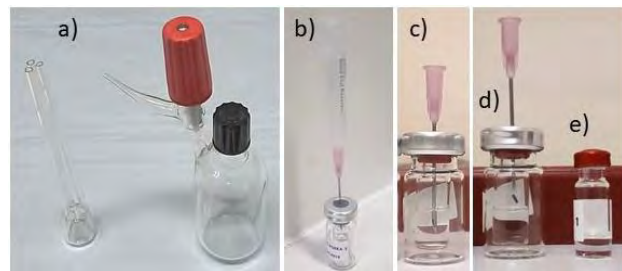


Figure 1. Different source designs that have been tested: a) Larger glass flasks, b) source design based on Shinohara et al. (2010), c) and d) adapted from Shinohara et al. (2010), with needle tip submerged and in headspace, respectively, and e) original design.

A few adaptations were then made to that design aiming to increase the emission rate. In the designs shown in Figure 1c and 1d (designs C and D, respectively), the use of the syringe and PE filter was discarded, and the needle size was increased (1,2 mm diameter, 40 mm length). In design C, the needle tip was submerged in the liquid tracer, but test results indicated lack of repeatability between sources (>50% std. error, n = 8). The design shown in Figure 1e (design E) consists of one single 1ml vial filled with ~0,5 ml of decane-D₂₂ capped with a metal cap, with the rubber stop substituted by a PE filter disk.

To compare the different source designs' emission rates, repeatability and leakage rates, gravimetry tests were executed using a micro-balance (accuracy: 0,01 mg). The weight loss of each source over time is then the source's emission rate. A stainless-steel climate chamber with inner volume of 112 dm³ (Figure 2) was employed to control T, RH and ACH in which the sources were kept in between the weighing moments.

In each gravimetric test, several replicate sources (a number varying from 4 to 12 simultaneous sources, depending on the tracer availability at each test) were kept in specified T, RH and ACH conditions inside the test chamber for a period of 5 up to 10 days, depending on the test. The replicates were weighed in intervals of 24 to 72h to observe the stability of their emission rates. The average weight loss of all the replicates was

then plotted against time, and the average emission rate of the sources in each test is equal to the slope obtained by linear regression.



Figure 2. Climate chamber used for source design testing.

A different set of chamber tests was then performed in order to evaluate the potential effect of different types of surfaces on the average tracer concentration reached after source placement. For these tests, one single source of decane-D₂₂ was placed inside the test chamber together with a sample of a surface made from a material commonly found in typical indoor environments. The materials were: an oak board (used in flooring), a coated plaster/gypsum board (used in walls) and a cloth/foam sample (a pillow). One control test was also performed in the absence of any surface sample. In each of these tests, one Radiello® passive sampler was placed inside the climate chamber to determine the average (equilibrium) tracer concentration resulting from the source plus surface interaction. Figure 3 shows the tests setup inside the chamber. Each test was carried out for 3 days, and the conditions in the chamber were constantly kept at $T = 15^{\circ}\text{C}$, $\text{RH} = 50\%$ and $\text{ACH} = 2,3 \text{ h}^{-1}$.

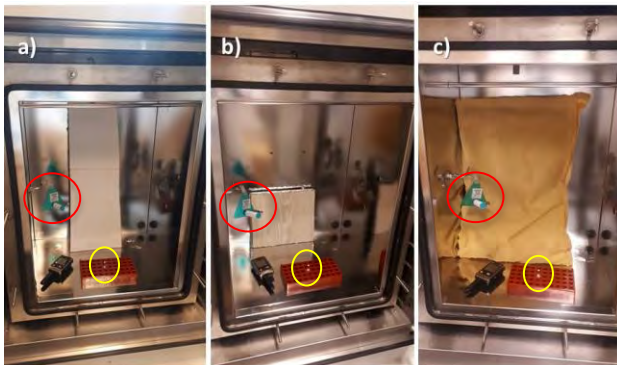


Figure 3. Setup of chamber tests designed to evaluate the effect of a) plaster/gypsum, b) wood and c) cloth/foam over the equilibrium tracer concentration. Tracer sources and samplers are circled in yellow and red, respectively.

RESULTS AND DISCUSSION

Selection of source design

Figures 4 and 5 show the average weight losses of the decane-D₂₂ source replicates over time for source designs D and E, respectively. In each figure, results for two different tests are shown, one under 25% RH and

the other under 75% RH. Moreover, both figures include the results of a leakage test for each type of source design.

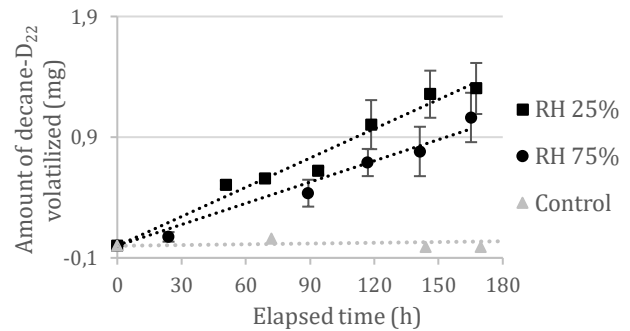


Figure 4. Results from two gravimetry tests comparing the average weight loss of the design D sources over time under 25% and 75% RH (both under constant $T = 23^{\circ}\text{C}$, $n = 6$).

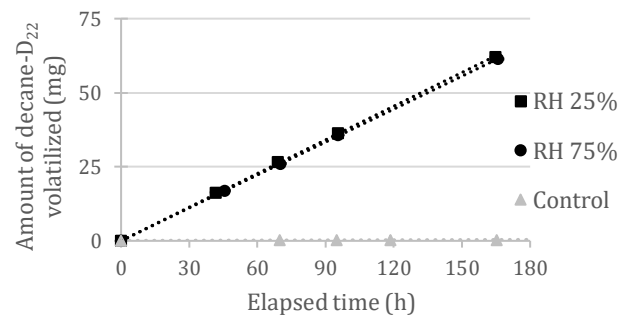


Figure 5. Results from two gravimetry tests comparing the average weight loss of the design E sources over time under 25% and 75% RH (both under constant $T = 23^{\circ}\text{C}$, $n = 6$).

Figure 4 shows that the weight loss of an unpierced source is insignificant over more than 10 days (last weighing result not shown), thus leakage of design D is negligible. Both tests showed a good linearity in the average measurements, indicating that Design D can keep a stable emission rate under different RH conditions. However, observing the linear regression slopes, the emission rate under drier conditions is slightly higher than under more humid conditions (decrease from ~ 8 to $\sim 6 \mu\text{g h}^{-1}$, corresponding to a relative difference of 27%), which indicates that the effect of RH over design D's emission rate cannot be neglected. The average repeatability error among the replicate sources was 11% for 25% RH and 25% for the 75% RH.

Similarly to what was observed with design D, results shown in Figure 5 indicate that leakage from a design E control source (i.e. source capped with a rubber stopper instead of a PE filter disk) is negligible for over 10 days (last weighing result not shown). Regarding the replicate sources participating in the emission tests, design E provided average emission rates two orders of magnitude higher than design D. Higher emission rates are desirable in order to ensure that the steady state tracer concentration during a TGT will be above the detection limit provided by the passive samplers. Linearity in both tests is almost perfect, indicating a high stability in the emission rates

provided by design E sources. Moreover, the effect of varying RH over design E's emission rate can be considered insignificant (decrease from $\sim 379 \mu\text{g h}^{-1}$ under 25% RH to $\sim 371 \mu\text{g h}^{-1}$ under 75% RH, corresponding to a relative difference of only 2%). The average repeatability error among the replicate sources was 3% for the test under 25% RH and 4% for the test under 75% RH. Therefore, based on the higher emission rate value, stability and repeatability, design E has been selected as the most suitable design source of decane-D₂₂ for the proposed TGT.

Effect of temperature and solvent level over source emission rate

Once the source design was selected, the next set of tests were intended to observe the effect of T over the source emission rates. Differently from RH, it is expected that T will have a significant impact on the tracer volatility, since it is known that the vapor pressure of a compound increases with increasing T, especially for VOCs.

A second factor that may have a significant impact over the source's emission rates is the level of liquid tracer inside the source. Figure 6 shows a source design E in detail.

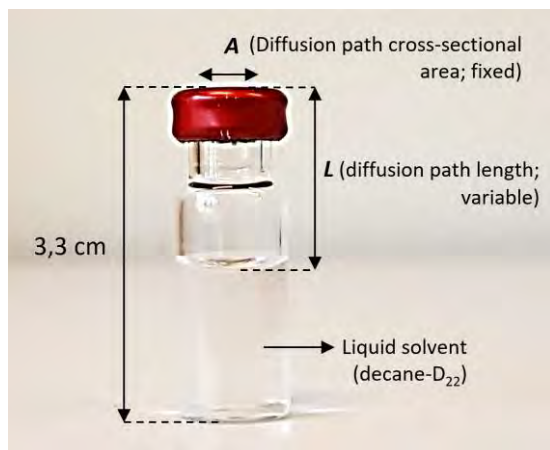


Figure 6. A design E source in detail, showing the tracer diffusion path.

According to Batterman et al. (2006), it is possible to use a simple Fickian diffusion model to predict the emission rate of a source from its geometry, as follows:

$$G = 3600C_s D_c \frac{A}{L} \quad (2)$$

In which G is the source emission rate (mg h^{-1}), A and L are the cross-sectional area (cm^2) and length (cm), respectively, of the tracer gas diffusion path (see Figure 5), D_c is the diffusion coefficient of the tracer gas in air ($\text{cm}^2 \text{s}^{-1}$), C_s is the saturated vapor concentration of the tracer gas (mg cm^{-3}), and 3600 converts from seconds to hours.

The model expressed in Equation 2 assumes that the tracer background concentration is negligible, that the tracer is promptly dispersed away from the source and that the tracer concentration in the headspace is saturated.

In order to apply Equation 2 to estimate design E sources emission rates, a few assumptions had to be made. Considering that decane-D₂₂ physio-chemical characteristics are the same as those of n-decane (except for the molar mass, which for decane-D₂₂ is $164,41 \text{ g mol}^{-1}$), the values of D_c and C_s at 20°C are set at $0,0574 \text{ cm}^2 \text{ s}^{-1}$ and $0,014 \text{ mg cm}^{-3}$, respectively, each calculated using a different online tool provided by EPA (2016) and Schmid (2019). Moreover, as observable in Figure 6, the value of A is not homogeneous through the whole length of L , i.e. the source's diameter (\varnothing) varies. To keep the calculations as simple as possible, an intermediate value of $\varnothing = 0,8 \text{ cm}$ was assumed. Still regarding the diffusion path, the length L includes both the inner headspace above the liquid solvent surface and the thickness of the PE filter disk in the metal cap (not visible in Figure 6). Although the thickness of the PE filter is of approximately $1,7 \text{ mm}$, the actual diffusion length imposed by it is much longer, due to its microporous nature, i.e. the tortuous path through the pores (average porosity $\sim 25 \mu\text{m}$). The exact diffusion length imposed by the PE filter disk (L_{PE}) can only be determined experimentally for each individual disk, but an average value of $L_{PE} = 1,3 \text{ cm}$ was adopted as an estimation for the additional diffusion path length in the theoretical calculations, using Equation 3 (Sigma-Aldrich, 2006).

$$G = 3600C_s D_c \frac{A}{L_{PE} + L} \quad (3)$$

Based on the aforementioned considerations and assumed values, the theoretical variation of G with L calculated with Equation 3 is shown in Figure 7.

Three sources with higher ($L = 2,1 \text{ cm}$), medium ($L = 2,5 \text{ cm}$) and lower ($L = 2,9 \text{ cm}$) solvent levels were simultaneously placed at constant 20°C and 0% RH to experimentally evaluate the validity of Equation 3 in predicting G given L and to investigate the effect of decaying solvent level over 10 days. The gravimetric results are also presented in Figure 7.

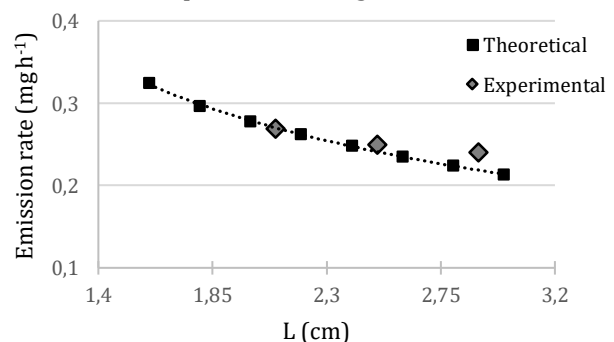


Figure 7. Tracer source emission rate as a function of the source's diffusion path length (L).

From the results shown in Figure 7, it can be seen that the actual effect of L over G is less prominent than theoretically predicted. The relative standard deviation (RSD) between the 3 sources was consistently only 5% in all weighing moments.

Moreover, the weight loss of all 3 sources over time was almost perfectly linear ($R^2 > 0,999$), demonstrating that the lowering in solvent level due to volatilization does not affect the source's emission rate for a period of 10 days (longer than the intended test application period - one week), regardless of the initial solvent level.

Figure 8 shows the graph of the emission rates achieved by sources with high ($L < 1,8$ cm) and low ($L > 2,5$ cm) solvent level as a function of the room temperature.

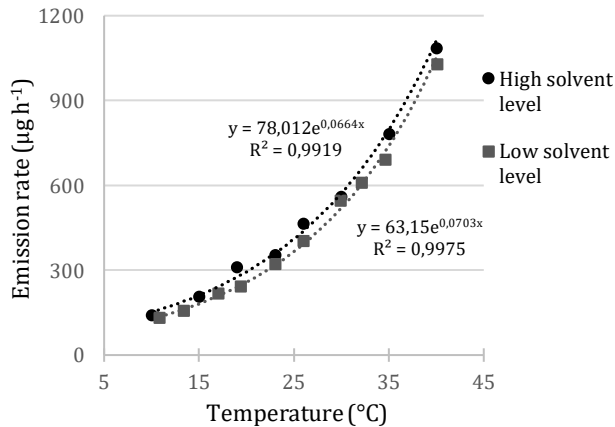


Figure 8. Tracer source emission rate as a function of room temperature. The regression equation and linearity are also presented.

As expected, in both cases the relationship is an exponential function, thus in real applications the exponential regression equations fit to the obtained datasets can be used to determine the emission rate from the average ambient temperature. However, the difference in solvent level did slightly influence the resulting exponential prediction curves. For a same given temperature, the two predicted values are ~10% different, with the relative difference decreasing with increasing temperatures. On the other hand, the individual sources at each temperature presented emission rates with excellent linearity ($R^2 > 0,99$) in both datasets, once again demonstrating that the decrease in solvent level due to normal volatilization does not significantly affect the source emission rate over time. Considering the results shown in Figure 7 and the slightly better fit of the exponential curve to the low-solvent-level dataset shown in Figure 8, it is indicated that a solvent level ranging between $L = 2,5$ to $2,9$ cm is applied in the sources for real-life field applications.

Tracer gas sorption to surfaces

After each material surface type was tested in the climate chamber (see Figure 3), one Radiello® passive sample was obtained, totalling 4 samples (one for each surface and one control test, no extra surface present). These samples were extracted with carbon disulphide (CS_2) spiked with a known amount of internal standard (2-fluorotoluene). The extracted samples were then analysed via gas chromatography coupled to mass

spectrometry. Figure 9 summarizes the results obtained after these 4 chamber tests.

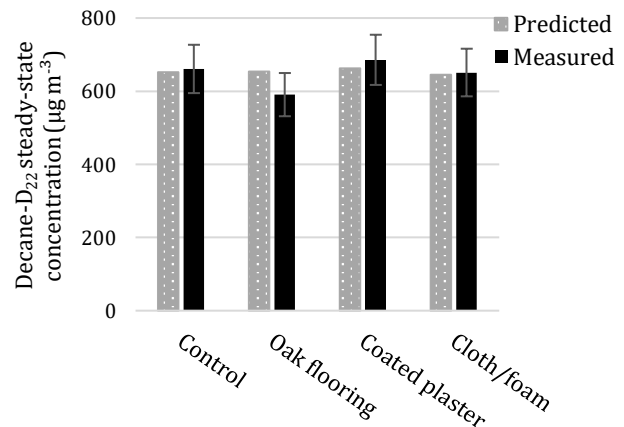


Figure 9. Results of chamber experiments testing decane- D_{22} sorption to different surfaces.

Besides the actual concentrations measured by the passive samplers in each test, Figure 8 also shows the predicted steady-state concentration of decane- D_{22} (C_s), calculated with Equation 4:

$$C_s = \frac{G}{qV_C} \quad (4)$$

Which is simply the rearrangement of Equation 1 to solve C_s instead of q (known and constant, in these tests), with V_C representing the inner volume of the climate chamber. For each test, G was measured gravimetrically.

The effect of the presence of each material over the steady-state tracer concentration can be evaluated by comparing the average concentration measured with the passive samplers to the expected concentration pre-calculated given the tests' conditions. In ideal conditions, no amount of tracer would "stick" to any surface, and the measured final concentrations would be exactly the same as predicted. The lower the actual measured concentration compared to the predictions, the "stickier" the material is to the tracer. As observable in Figure 9, the measured concentrations were in fact very close to the calculated predictions, and in some cases even somewhat higher (which is most likely due to small fluctuations in the inner chamber conditions during the test, as well as to contrasting conditions during weighing moments/chamber door openings that cannot be accounted for in the calculations). Even the effect of the oak flooring sample, the most prominent among the 3 tested materials, can be considered insignificant considering the intrinsic error of the Radiello® samplers (of 10%, error bars shown in Figure 9). Therefore, the effect of decane- D_{22} sorption to surfaces is considered to be negligible for the applications intended in this study.

CONCLUSIONS

The final goal of this research is to provide new adaptations to the most used method to measure

ventilation rates in field, i.e. the TGT, as a way of making it so practical and simple that it ultimately encourages researchers, contractors and building owners to include actual ventilation measurements in their routine IAQ assessments. The present paper focuses specifically on describing the process of selecting a new tracer gas and of designing an adequate tracer source. From the data gathered via literature review and lab experiments, decane-D₂₂ is considered to be a suitable tracer substance to substitute the currently employed tracers with high global-warming potential. The passive source design ultimately selected after lab testing (design E) provides stable and repeatable decane-D₂₂ emission rates under standard temperature. Varying RH and ACH were shown to not affect the tracer emission rate. The effect of the liquid solvent level inside the source over the tracer emission rate was only barely noticeable. Nonetheless, a strict range of adequate solvent level is suggested. The selected tracer was also shown not to adhere/absorb significantly to surfaces. Additionally, a consistent exponential curve was derived for determining the source emission rate from the room temperature. These positive results are a good indication of the real-life applicability and adaptability of the new adapted TGT. Field test applications are currently being performed for validating and further confirming the new TGT as an adequate alternative to currently performed tests.

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