



# International comparison of key volatile organic components in indoor air

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## ABSTRACT

An international comparison was coordinated by the National Physical Laboratory to assess the analytical capabilities of laboratories for measuring ten selected volatile organic compounds. These were chosen to represent a wide range of non-methane hydrocarbons, terpenes and oxygenated hydrocarbons, which pose a significant risk to public health from their presence in indoor environments from emissions by building and consumer materials. The components were loaded onto sorbent tubes and distributed to twenty-four participating laboratories for analysis. It was found that laboratories predominantly reported analytical results that were greater than the reference value, however the majority of results were closer to the reference value when compared to the previous comparison carried out in 2010.

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## 1. Introduction

It has been well documented that air quality influences human health. Poor air quality has been linked to higher mortality rates, and other adverse health effects, such as bronchitis and cardiovascular problems and the phenomenon known as Sick Building Syndrome [1,2]. There is significant work being carried out globally to ensure that air quality is being monitored and regulated to minimise negative effects on public health [3–5].

While there has been extensive research to study the impact of components in outdoor air on public health, measurements of indoor air quality have been a lower priority. Currently in the European Union there are three obligatory national schemes for assessing emissions in indoor air from construction products, the most well-known being the German AgBB evaluation scheme, as well as a number of voluntary schemes. In the forthcoming years it is likely that more regulations will be introduced. According to

the US Environmental Protection Agency (EPA), indoor air can be two to five times more polluted than outdoor air [6]. As the public are spending increasing lengths of their time indoors, it is necessary that careful monitoring is implemented and to achieve this, the development of a traceable measurement infrastructure is required. This is of increasing importance within the construction of new buildings and there are international and European Union Construction Products Directives including EN ISO/IEC 16000-11:2006 [7] and CEN/TC 351 (document number 0588) [8] associated with these and other air pollutants. With continual improvements being made to seal buildings in an effort to make them more energy efficient, the overall ventilation and air exchange rates with the outside have reduced, leading to heightened concentrations of pollutants trapped indoors [9].

It is essential that laboratories carrying out the analysis of volatile organic compounds (VOCs) found in indoor air ensure that results are comparable and traceable. Proficiency testing schemes are one way of showing compliance with quality control procedure and demonstrating comparability [10,11]. One such scheme for assessing

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global laboratory performance in air quality analysis is the Ambient, Indoor, Workplace Air and Stack Emissions Proficiency Testing Scheme (AIR PT Scheme) [12]. This is a combination of the former Workplace Analysis Scheme for Proficiency (WASP) [13] and Stack Emissions Proficiency Testing Scheme (STACKS) [14]. However, while such schemes provide an insight into the performance of analytical laboratories for measuring routine components, such as benzene, studies of components with emerging measurement requirements for indoor air quality are scarce.

An international comparison was coordinated by the National Physical Laboratory (NPL), in which ten selected components commonly found in indoor air from construction material emissions, were either gas or liquid loaded onto sorbent tubes and sent for analysis to each of the participating laboratories. The exercise was aimed at laboratories (some of which are ISO 17025 accredited [15]) undertaking the analysis of VOCs from air samples collected on sorbent tubes and analysed using thermal desorption techniques as detailed in ISO 16017-1 [16] and 16017-2 [17]. This comparison provides a means of assessing the analytical capability of each laboratory [18,19]. The results of the comparison are presented and contrasted to a similar comparison carried out in 2010 [10].

## 2. Experimental

To keep anonymity each participant and sample set was ascribed a letter A to X, and the assignment was only known to the coordinators at NPL. Participants were supplied with Silco-treated Tenax<sup>®</sup> sorbent tubes: one blank and three loaded (despatched in packing containers) with benzene, toluene, butyl acetate, hexanal, *m*-xylene,  $\alpha$ -pinene (both optical isomers were included at approximately an even ratio), styrene, 1,2,3-trimethylbenzene, 2-ethyl-1-hexanol and 1-methyl-2-pyrrolidone. Four of the components selected (benzene, toluene, butyl acetate and 2-ethyl-1-hexanol) featured in a comparison organised by NPL in 2010. In addition to the components included in the 2010 comparison, six additional components were chosen that are commonly found in indoor air from building emissions but are not frequently analysed. Analysis of blanks confirmed that packaging and transport conditions did not introduce material onto the sorbent tubes.

2-Ethyl-1-hexanol and 1-methyl-2-pyrrolidone were liquid loaded due to their low vapour pressures. This was done prior to the gas loading of the other components, sorbent tubes were loaded with 2-ethyl-1-hexanol and 1-methyl-2-pyrrolidone from a liquid reference standard following a similar method to that adopted by Martin et al. [20]. A nominal 5  $\mu$ L aliquot of a nominal 40  $\mu$ g L<sup>-1</sup> stock solution containing both components was injected from a 10  $\mu$ L syringe through a modified injection port onto each sorbent tube. After each injection, compressed air scrubbed by a filter was passed over the sorbent tube for a minute at a flow rate of approximately 150 mL min<sup>-1</sup> to remove the solvent. The 10  $\mu$ L dispensing syringe was weighed before and after injection to determine the loaded mass transferred onto each sorbent tube. The volume of the syringe was set by a Chaney adaptor to improve repeatability.

The remaining eight components were loaded from a Primary Reference Gas Mixture (PRGM), gravimetrically prepared and validated at NPL. The composition of the PRGM is shown in Table 1. The PRGM was passed over each sorbent tube in turn at a nominal flow rate of 10 mL min<sup>-1</sup> for 15 minutes. A minimised dead volume connector and a low volume restriction device (used to control sample flow) were employed. Each day the system was purged for two hours before tube loading commenced. The other end of the sorbent tube was connected to a flow meter (BIOS), which was used to record the flow during each tube loading. In total ninety-two sorbent tubes were loaded over four days. The mass loading of all of the eight components was determined using Eq. (1), where  $m$  is the mass of the analyte loaded (g),  $f$  is the PRGM flow rate (L min<sup>-1</sup>) at STP,  $t$  is the loading time (min),  $V_m$  is the molar volume of an ideal gas at standard temperature and pressure (L mol<sup>-1</sup>),  $x$  is the amount fraction of the component in the PRGM (mol mol<sup>-1</sup>) and  $M_w$  is the molecular weight of the component (g mol<sup>-1</sup>).

$$m = \frac{ftxM_w}{V_m} \quad (1)$$

A set of gravimetric liquid standards, containing all of the ten VOC components, were prepared at nominal concentrations of 20, 40 and 60 g L<sup>-1</sup> in methanol ( $\geq 99.9\%$ , Sigma Aldrich). These were diluted with the same methanol to produce solutions with nominal concentrations of 60, 100, 200, 400, 600 and 1000  $\mu$ g L<sup>-1</sup>. Aliquots of 1  $\mu$ L of the liquid standards were injected onto Silco-treated Tenax<sup>®</sup> sorbent tubes (as previously described for 2-ethyl-1-hexanol and 1-methyl-2-pyrrolidone) and analysed using Thermal Desorption Gas Chromatography (TD-GC) Flame Ionisation Detection (FID) and Mass Spectroscopy (MS) as outlined in 16017-2 to create a calibration curve. This was used to validate the gravimetric loadings of a sample of eleven tubes taken from the batch loaded for the comparison. The batch of eleven tubes comprised the first and last tube loaded on each day, as well as an additional tube from each of the first three days. These measurements were also used to interrogate the homogeneity of the batch. For most components the standard deviation for analysis of the batch of eleven tubes was less than 2%.

An indoor air method was created where the Tenax<sup>®</sup> sorbent tubes were desorbed for 15 minutes at 275 °C. The system was dry purged for three minutes and then

**Table 1**

The amount fraction of components in the primary reference gas mixture used for gas loading.

Component	$x$ (nmol mol <sup>-1</sup> )
Benzene	248.6
Toluene	262.8
Butyl acetate	253.3
Hexanal	242.6
<i>m</i> -Xylene	250.2
$\alpha$ -pinene	246.3
Styrene	254.5
1,2,3-Trimethylbenzene	237.7
Nitrogen	Balance

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