

# Method development for measuring volatile organic compound (VOC) emission rates from spray foam insulation (SPF) and their interrelationship with indoor air quality (IAQ), human health and ventilation strategies

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## ABSTRACT

The polyurethane foam industry is projected to reach a worldwide value of up to \$74bn by 2022 and with airtightness of new and retrofitted properties continually increasing, an important question arises: what is the impact of these materials on the indoor air quality (IAQ), occupants' health and indoor environment?

As the foams are made in-situ through an exothermic reaction between two chemical mixtures (side A and side B), volatile organic compounds (VOCs) are emitted during their application and curing process. Current research, commercial practices and governmental advice suggests that emissions decrease over time and 8-24 h after application are usually sufficient for residents to return safely to their properties. However, there is still a lack of case studies and a fundamental absence of robust analysis on how ventilation strategies affect long term off-gassing rates and chemical emission quantities. The emission rates from SPF materials could have a direct impact on IAQ if they exceed the occupational exposure rates recommended by NIOSH, or other professional associations. But the difficulty in recording these emission rates is evident as there is still a lack of an international standard for their quantification.

To address this issue, we have developed an analytical methodology for measuring some of the composition materials of the foams and residual products associated with their application. The experiment consisted of two stages- active air sampling of spray foam emissions and spiking desorption tubes with a standard solution in order to develop calibration curves. The solution included SPF compounds, or by-products from their application, associated with possible acute impact on health: 1,4 dioxane, chlorobenzene, dibutyltin dilaurate, triethyl phosphate and bis(2-dimethylaminoethyl)ether. We managed to detect five of the chemicals of interest through air sampling and produce calibration curves for 1,4 dioxane, chlorobenzene and triethyl phosphate, which would allow us to quantify the emission rates at the next stage of research.

The results of the experiments successfully demonstrated proof of concept quantitative methodology for the compounds of interest. With further research and experiments, this technique has the capacity to be developed into an international standard for measuring VOCs from spray foam emissions and other buildings products. This would provide scientists and industry professionals with the tools to further develop retrofit and ventilation strategies in order to provide healthier buildings.

## KEYWORDS

Volatile organic compounds, spray polyurethane foam, indoor air quality, human health, ATD-GC-MS

## 1 INTRODUCTION

With people spending up to 90% of their time indoors, measuring the effect of building material emissions on human health and indoor air quality has quickly become a critical issue (Seddon, 2015) (Lai et al. 2004). Polyurethane spray foam (SPF) sales have topped £1bn in 2015, although the factors affecting their performance are widely unknown (Bomberg, 1998). Only in recent years, their impact on human health and indoor air quality (IAQ) has become an imminent topic for research with industrial and governmental bodies outpacing academic research (ASTM International 2017). The ASTM International consensus on standards for chemical emissions from spray foams (2017) represents 13 papers and signals the need for further understanding of the topic. According to a recent market study report (Markets and Markets, 2016) (Lucintel, 2017) the polyurethane foam industry will grow to \$60.5-74.24bn by 2022, therefore there is an urgent demand for scientific analysis of the impact of these materials on the indoor air quality, human health and effectiveness of different ventilation strategies.

Personal exposure levels to chemical compounds are affected by a mixture between indoor and outdoor pollutants rather than a single source. In order to determine more accurately the effect that building products have on human health, the emission rates of their compounds, or by-products associated with their application, must be recorded. Modern SPF insulating products are produced in-situ through a chemical exothermic reaction between the A-side component (usually MDI<sup>1</sup>, pMDI<sup>2</sup> or TDI<sup>3</sup>) and the B-side component (polyols, fire retardant, catalysts, blowing agents and surfactants). Exposure to the elements of the A-side during application (Crespo & Galan, 1999), its concentration (Lesage et al. 2007) and its impact on human health (Kupczewska-Dobecka et al. 2012) have been measured by using a variety of techniques and methodologies. These include impinger sampling with high-performance liquid chromatography and isocratic reverse phase analysis (HPLC/RP, isocratic) (Schlecht & Cassinelli 1998), impinge sampling with liquid chromatography coupled with ultraviolet detection (LC-UV) (Crespo & Galán 1999), glass fibre filter followed by liquid chromatography coupled with fluorescence detection (LC-FL). The B-side has been primarily analysed with automated thermal desorption-gas chromatography-mass spectrometry (ATD-GC-MS) for informing re-entry times after installation (Wood, 2017), measuring emissions of flame retardants and blowing agents (Poppendieck et al. 2016) (Sleasman, et al., 2017) and VOC emissions testing under different application conditions (Won, et al., 2017).

Our experimental setup was designed to contribute to the development of international analytical standards and aimed to provide scientific evidence for the future establishment of voluntary and mandatory protocols for measuring emission rates from building products. The objectives of the experiment were:

- To determine robustness of ATD-GC-MS analytical method for qualitative and quantitative analysis of volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs) and very volatile organic compounds (VVOCs) from spray foam insulation products
- To examine which compounds and by-products associated with SPF could be detected by using Carbopack-B sorbent tubes and to establish calibration curves for some of the chemicals associated with acute impact on human health

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<sup>1</sup> 4,4' Methylene diphenyl diisocyanate, CAS #101-68-8

<sup>2</sup> Polymeric MDI, CAS # 9016-87-9

<sup>3</sup> Toluene diisocyanate, CAS #584-84-9

## 2 METHODS

The experiment was consisted of two stages: active air sampling of VOC emissions and spiking thermal desorption tubes with a solution consisting of SPF compounds and residual products from its application.

### 2.1 Spray foam active air sampling

A sample of a closed cell spray foam was provided by Q-Bot Ltd (London, United Kingdom). The sample was sprayed directly in a 568 mL expanded polystyrene cup by using remotely controlled robots. The conditions under which the foams were created were not recorded, but it was confirmed that standard procedures as per the manufacturer guidelines for the product application have been followed. The SPF was made on the 17<sup>th</sup> of January 2017 and stored in a sealed vacuum bag at room temperature (18-23 °C) in the laboratory for a period of 129 days before being sampled thus providing sufficient time for the foam to cure.

The sample was then cut in a fume hood into ~3-5 cm pieces with a disposable steel knife in order to minimise airborne contamination and following best practice guides (ACC Spray Foam Coalition 2016). The pieces were placed in a 1-L glass bottle for a period of 1 h before sampling. The glass bottle was conditioned in an oven for 24 h at 140 °C prior to the experiment to ensure that there was no contamination. The weight of the sample was not recorded as the purpose of this stage of the experiment was to obtain qualitative results only.

The experimental setup for sampling can be seen in Figure 1. The glass bottle had an inlet and an outlet, to which Carbopack-B tubes were connected. The incoming air (blue in Figure 1) was filtered through the first tube and then travelled to the bottom of a plastic tube in order to ensure even airflow throughout the vessel. The second Carbopack-B tube was connected to a low flow air pump which was set to 40 mL/min ( $\pm 5\%$ ) and air was extracted from the headspace for 44 min (red in Figure 1). For future experiments where quantitative results will be sought a second extraction tube will be coupled with the first one in a series. If any of the chemicals are found in the second tube, the results would not be used and either the extraction time would need to be adjusted or the model of sorbent within the tubes would need to be altered. Prior to sampling the tubes were conditioned at 50 mL/min and heated to 350 °C for 60 min. The equipment and process of analysis was fully automated with a central software and the experiment was undertaken with controlled flow and temperature.

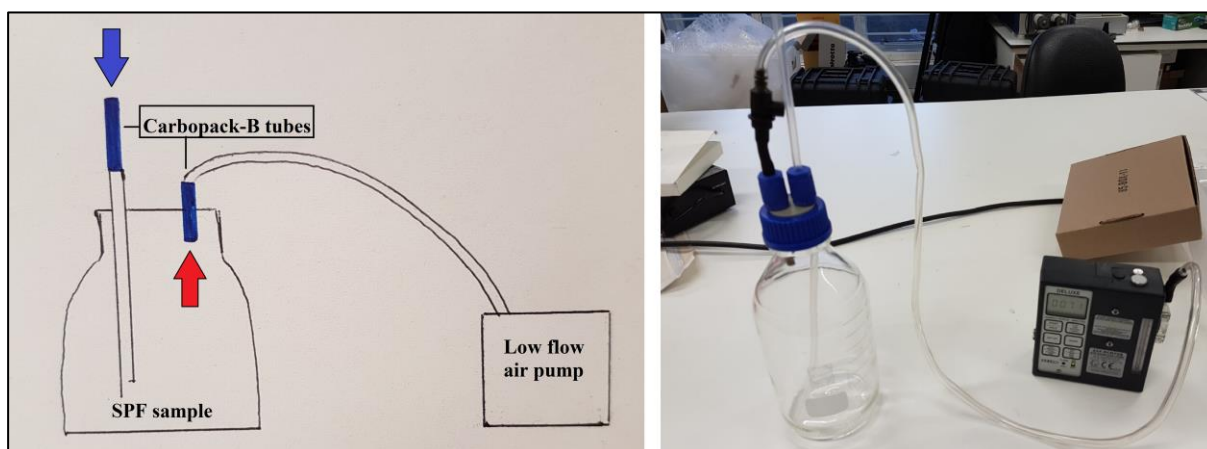


Figure 1. Experimental setup for VOC air sampling from SPF products

## 2.2 Desorption tube spiking

“Spiking” of a desorption tube consists of injecting a small quantity of a prepared chemical solution or standard directly onto the top mesh of the tube. We created a bespoke standard for the purpose of the analysis. Through previous work with solid-phase microextraction-gas chromatography-mass spectrometry (SPME-GC-MS) and literature review, it was determined that ATD-GC-MS would be more suitable for the analysis of the B-side and residual products of the foam (Schlecht & Cassinelli 1998; Creely et al. 2006; ASTM International 2017).

We selected chemicals that could possibly have a negative impact on health. The solution was made by using a micropipette and injecting individual chemicals into a 10-ml glass volumetric flask with quantities as per Table 1. The rest of the flask was filled with HPLC-grade 99.9% methanol.

Ten tubes were injected with 1  $\mu\text{l}$ , 5  $\mu\text{l}$ , 10  $\mu\text{l}$ , 15  $\mu\text{l}$  and 20  $\mu\text{l}$  of the solution respectively, to provide the calibration range as per Table 1, with an electronic pipette. Two tubes were used for each quantity in order to deliver average values and derive calibration curves. As per the air sampling method, the tubes were conditioned prior to the experiment with 50 mL/min nitrogen at 350  $^{\circ}\text{C}$  for 60 min. Once the tubes were spiked they were immediately placed within the ATD carousel and the analysis was started in order to ensure accurate results.

Table 1. Standard solution composition

Chemical	CAS Number	Individual weight in vial (mg)	ng/ $\mu\text{l}$	Calibration range (ng)
1,4 dioxane	123-91-1	0.505	50.5	50.5-1010.0
Chloro-benzene	108-90-7	0.525	52.5	52.5-1050.0
Dibutyltin dilaurate	77-58-7	0.405	40.5	40.5-810.0
Triethyl phosphate	78-40-0	0.754	75.4	75.4-1508.0
N,N,N',N' – Tetramethyl-2,2'-oxybis(ethylamine)	3033-62-3	0.421	42.1	42.1-842.0

## 2.3 Instruments and analytical conditions

The samples were collected on Carboxpack-B desorption tubes, which have an analyte volatility range from n-C5 to n-C12 and are suitable for a wide range of VOCs. They are compatible for both active sampling and direct liquid injections. The tubes were placed in a carousel on a Perkins Elmer TurboMatrix 650 automated thermal desorption kit connected to a Perkins Elmer 500 gas chromatograph coupled with a Perkin Elmer Clarus 560D mass chromatograph. The column used was a 60m x 0.25mm x 1.5  $\mu\text{m}$  VOCOL fused silica capillary column with helium used as a carrier gas.

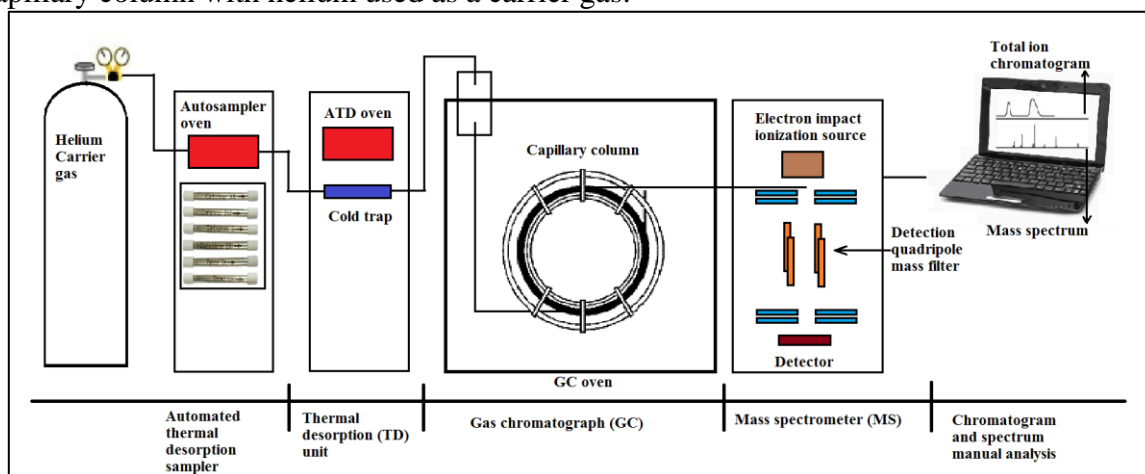


Figure 2. Experimental setup of analytical methodology

This technique allows for a non-destructive headspace analysis of the VOCs emitted by the insulation materials. Albeit in our case we cut the sample in order to exacerbate the emission rates and confirm the robustness and detection limits of the analytical methodology.

The method summary can be found in Table 2.

Table 2. ATD-GC-MS system settings

<b>ATD</b>	<b>Air sampling</b>	<b>Tube spiking</b>
<b>Purge</b>		
Prepurge time	1 min	1 min
Trap in line	No	No
Split	On	On
Flow rate	25 mL min <sup>-1</sup>	25 mL min <sup>-1</sup>
<b>Tube desorption</b>		
Time	8 min	8 min
Temperature	300 °C	300 °C
Split	Off	Off
<b>Trap desorption</b>		
Trap low temperature	-10 °C	-10 °C
Trap high temperature	330 °C	330 °C
Trap hold time	5 min	5 min
Split	On	On
Trap heating rate	40 °C sec <sup>-1</sup>	40 °C sec <sup>-1</sup>
Split flow rate	25 mL/min	35 mL/min
<b>Split ratios</b>		
Inlet	32.5:1	No split
Outlet	26.3:1	26.3:1
Total	58.8:1	26.3:1
<b>Other</b>		
Flow path temperature	250 °C	250 °C
GC cycle time	60 min	120 min
<b>GC</b>		
Helium Flow	1mL min <sup>-1</sup>	1mL min <sup>-1</sup>
<b>Temperature Profile</b>		
Initial Temperature	35 °C (hold 5 min)	50 °C (hold 5 min)
Ramp	10 °C min <sup>-1</sup>	10 °C min <sup>-1</sup>
Second Temperature	200 °C (hold 10 min)	100 °C (hold 0 min)
Ramp		7 °C min <sup>-1</sup>
Third Temperature		200 °C (hold 0 min)
Ramp		2 °C min <sup>-1</sup>
Final temperature		220 °C (hold 25.7 min)
Total run time	31.5 min	60 min
<b>MS</b>		
MS Ionization Mode	E+	E+
MS Inlet Temperature	200 °C	200 °C
MS Source Temperature	180 °C	180 °C
Mode	Scan	Scan
Mass Scan Range	45-300	45-550

### 3 RESULTS

The purpose of these experiments was to obtain qualitative results and establish calibration curves in order to deliver quantitative results in future research.

#### 3.1 Active air sampling chromatogram analysis

The experiment was successful and the chromatograms clearly showed that a number of VOCs were detected from the spray foam. The chromatograms were consistent and clearly resolved peaks could be analysed and identified. There were generally no unusual peak shapes, apart from peak tailing and background noise towards the end of the run. This could have been a sign that the detector temperature of the GC-MS was too low for the samples tested (Prichard, 2003).

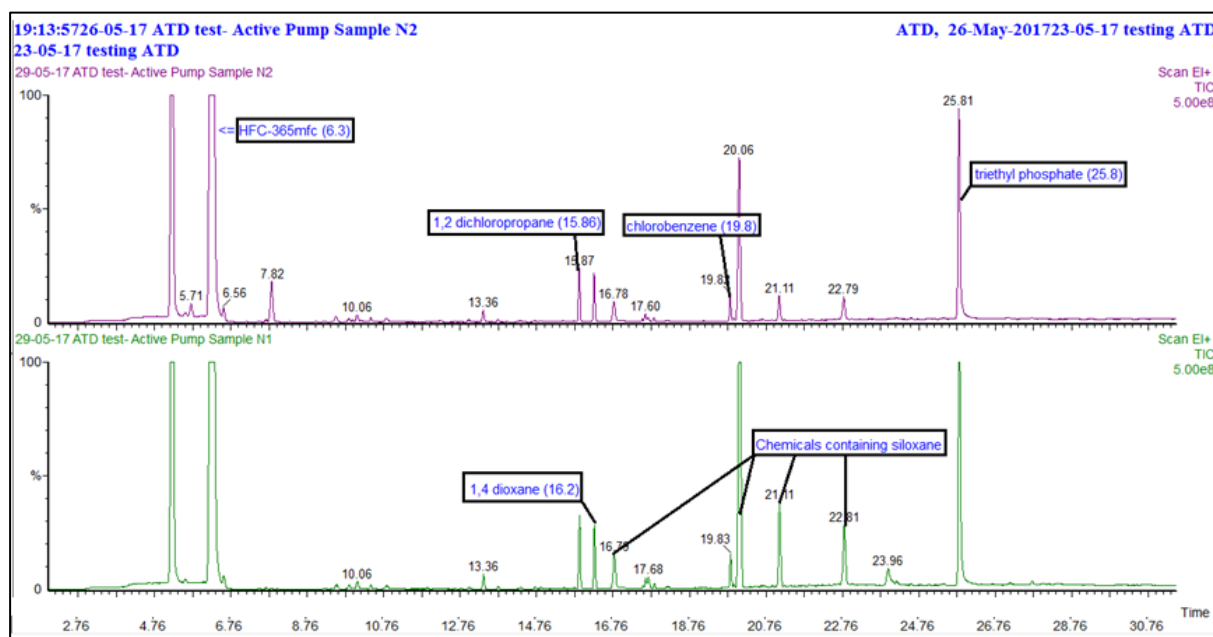


Figure 3. Air sampling chromatograms

The chromatograms displayed a good range of compounds, which was expected due to the complex structure of SPF products and the high sensitivity of the equipment. In order to detect the compounds, manual analysis of the peaks was undertaken through single ion monitoring.

Each of the peaks was cross-referenced to the NIST library database for qualitative assessment. The results were analysed by cross-checking what the peaks most likely are, based on NIST hits, and through searching for the composition chemicals of the foam and expected by-products found from previous analysis (Sleasman, et al., 2017) with the results summarised in Table 3. The ones in red could not be found in the NIST library. Those highlighted in yellow are compounds present in the NIST library, but could not be found during the analysis. The green ones are the detected compounds, which have been successfully matched to their mass spectra within the library.

Table 3: Table Caption

Composition	Target compound	CAS Number & Boiling Point	Product Function	Health impact labels <sup>4</sup>
Side A	Diphenylmethane-4,4'-diisocyanate (MDI)	101-68-8 ; 314 °C	Thermal Insulation (once blown)	
Side B	Dibutyltin dilaurate	77-58-7; 204 °C	Catalyst	
	Triethyl Phosphate	78-40-0; 215 °C	Flame retardant	
	1,1,1,1,3,3-pentafluorobutane (HFC-365mfc)	406-58-6; 40 °C	Blowing agent	
	N,N,N',N'-Tetramethyl-2,2'-oxybis(ethylamine)	3033-62-3; 189 °C	Blowing catalyst	
	Tris(2-chloro-1-methylethyl)phosphate	13674-84-5; 235 °C	Flame retardant	
	N,N-bis[3-(dimethylamino)propyl]-N',N'-dimethylpropane-1,3-diamine	33329-35-0; 297.7 °C	Catalyst	
Residual products	1,4 dioxane	123-91-1; 101 °C	By-products	
	1,2-dichloropropane	78-87-5; 95.5 °C		
	Chlorobenzene	108-90-7; 132 °C		

With the Carbopack-B tubes, five of the chemicals were recorded by using the setup as per Table 2. The detected chemicals had boiling points between 40-215 °C, which demonstrates the effectiveness of this method for a wide range of volatile compounds.

The most distinctive emission, associated with the highest peak, was the blowing agent HFC-365mfc. This is expected as the foam was cut, therefore releasing the blowing agent which was trapped within the closed cell structure of the foam. Future experiments would have to make bespoke vessels or emission chambers to ensure that only the surface emission are taken into account in testing. It must be pointed out that all of the residual elements were found even after the foam had cured for 129 days. This leads to the conclusion that the composition chemicals of the foam react with the indoor air (even filtered) and thus the residual compounds are released.

### 3.2 Spiked tubes analysis

The dibutyltin dilaurate and the N,N,N',N'-tetramethyl-2,2'-oxybis(ethylamine) could not be found on the chromatograms when using Carbopack-B tubes. The other three chemicals (1,4-dioxane, chlorobenzene and triethyl phosphate) registered clearly resolved peaks with a linear progression depending on the amount injected onto the tubes.

<sup>4</sup> All figures sourced from PubChem database and based on GHS classification

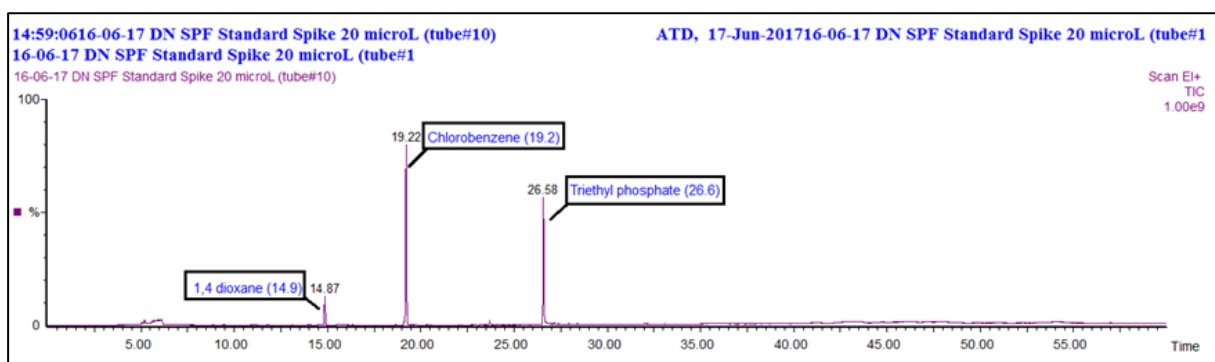


Figure 4. Spiked tubes chromatograms

Chromatographic peak areas were derived by integrating the peak area for each compound. The results were plotted against the known quantity of each material in order to form the calibration curves as per Figure 5. The calibration curves demonstrated a continuous linear increase when the average results for each point were calculated. The standard errors in slope and intercept have been calculated with the full data set. It should be noted that the linearity range has not been exceeded and further experiments will be conducted with higher amounts of the analytes to do so. Once the calibration curves are further established, we will be able to quantify the emission rates.

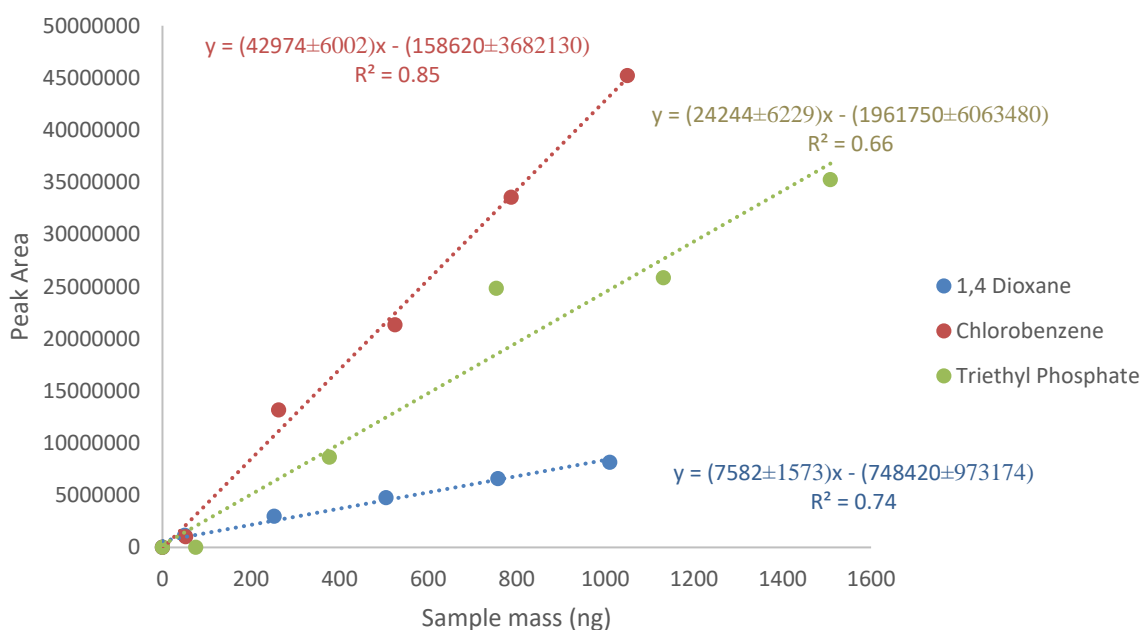


Figure 5. Calibration curves for 1,4-dioxane, chlorobenzene and triethyl phosphate

#### 4 DISCUSSION

The success of both experiments clearly demonstrates the potential of the technique and its repeatability in detecting the chemical compounds from SPF products and by-products released from them. As the isocyanates (MDI) are semi-volatile organic compounds, they demonstrate a high sink effect and due to their high boiling point, they could not be volatilised for analysis. They are highly unstable particles and adhere easily to surfaces therefore making them difficult to detect (Crespo & Galán 1999; Puscasu et al. 2015; Lesage et al. 2007; Kupczewska-Dobecka et al. 2012) and therefore for this reason ATD-GC-MS is not a suitable methodology for their analysis.



There were siloxane elements detected in all tests, which are most likely released from the column as they are also consistently present in the blank samples in very small quantities. It must be noted that some of the other compounds from SPF products have been successfully quantified through the use of Tenax-TA tubes, emission chambers and ATD-GC-MS such as tris(1-chloro-2-propyl)phosphate (TCPP) (Poppendieck et al. 2016), therefore reinforcing the case for the analytical methodology as proposed here.

1,4-Dioxane and chlorobenzene are known to cause headache, numbness and nausea (Agency for Toxic Substances and Disease Registry 1990), eye and nose irritation (Agency for Toxic Substances and Disease Registry 2012). These are one of the common health impact issues recorded after improper installation of SPF insulation products (Huang & Tsuang 2014). The results prove that the residual products from SPF can persist in the air if the foam is damaged and it is crucial that the emission rates are quantified in order to devise ventilation strategies to reduce their impact on IAQ and human health. Further investigation is needed in order to determine which chemicals exactly might be responsible for the negative side effects experienced under faulty applications. In addition, there is no internationally established “faulty application of spray foam insulation materials” definition, but only guidelines for environmental conditions during application and mechanical setup of the spraying equipment.

## **5 INTERRELATIONSHIP OF VOC, IAQ AND VENTILATION STRATEGIES**

Indoor air quality (IAQ) is directly linked with the health and comfort of the building occupants and is determined by the range of indoor and outdoor pollutants (Crump et al. 2009). The volatile organic compounds associated with the application of SPF materials, as per Table 3, could negatively impact IAQ unless robust ventilation strategies are in place to purge the emissions. Understanding how the risks of building product off-gassing can affect the IAQ parameters is crucial as air-permeability standards for new and retrofitted buildings is continually decreasing. For new build dwellings in the UK to comply with Building Regulations Part L1A, they usually need to achieve an air-permeability of  $5 \text{ m}^3/(\text{h}\cdot\text{m}^2)$  or lower. As air-tightness of buildings increases, the need to have robust ventilation strategies, natural or mechanical, in order to provide healthy IAQ and environment is becoming more apparent and necessary.

Higher ventilation rates are required immediately after application as the emission rates from SPF materials are usually the highest immediately after application (Duffy & Wood, 2017). But so far the focus of research has been on short term analysis such as re-entry times (Wood, 2017), VOC emissions under different applications (Won, et al., 2017) and negative impact associated with faulty SPF applications (Huang & Tsuang 2014). The potential long-term impact of these buildings materials and what ventilation strategies should be in place throughout the lifecycle of the building, in order to provide healthy IAQ, is still widely unknown. It is therefore crucial that an international standard for measuring emission rates from SPF materials is adopted in order for further long-term research on the topic to be undertaken.

## 6 CONCLUSIONS

The method of SPF VOC analysis by using ATD-GC-MS has proven to be appropriate with the potential to be developed into an international measurement standard. Carbopack-B tubes proved efficient in detecting five of the chemicals associated with SPF, which could be a cause for some of the common health impact issues. In addition, calibration curves were developed for 1,4-dioxane, chlorobenzene and triethyl phosphate, which could be used for quantification of the emission rates.

Unfortunately, the technique is unsuitable for measuring isocyanates due to their high boiling point, sink effect and instability (Streicher et al. 2000; Sebroski et al. 2012; Lesage et al. 2007).

The industrial development and growth of the SPF sector has steadily outgrown the research undertaken on its interrelationship with ventilation rates, IAQ and human health. It is therefore essential that this analytical methodology is further developed in order for an international standard to be established for measuring VOCs from these building products.

In our future research, we will further establish the methodology and develop the field through the following planned work:

- Other desorption tubes will be used in order to detect all chemicals associated with SPF application, apart from isocyanates, with ATD-GC-MS. This will allow for holistic studies to be undertaken with the same technique and enhance the speed with which an international standard is established.
- Long-term emission (>1-2 years) testing must be undertaken in order to determine how the chemicals might affect IAQ throughout the SPF lifecycle, rather than simply during and shortly after the installation process
- Real life case studies must be used to test various ventilation strategies and systems during and post-application of the foams. Through this data, the effectiveness of various natural or mechanical ventilation strategies will be tested in order to ensure that retrofitting measures, and new build applications, with SPF do not compromise the healthy IAQ.

## 7 ACKNOWLEDGEMENTS

The authors are grateful to Mark Underhill for technical support and to Q-Bot Ltd. for providing the samples.

The research has been funded through the EPSRC Centre for Doctoral Training in Science and Engineering in Arts, Heritage and Archaeology (SEAHA) and is partially funded by Q-Bot Ltd.

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