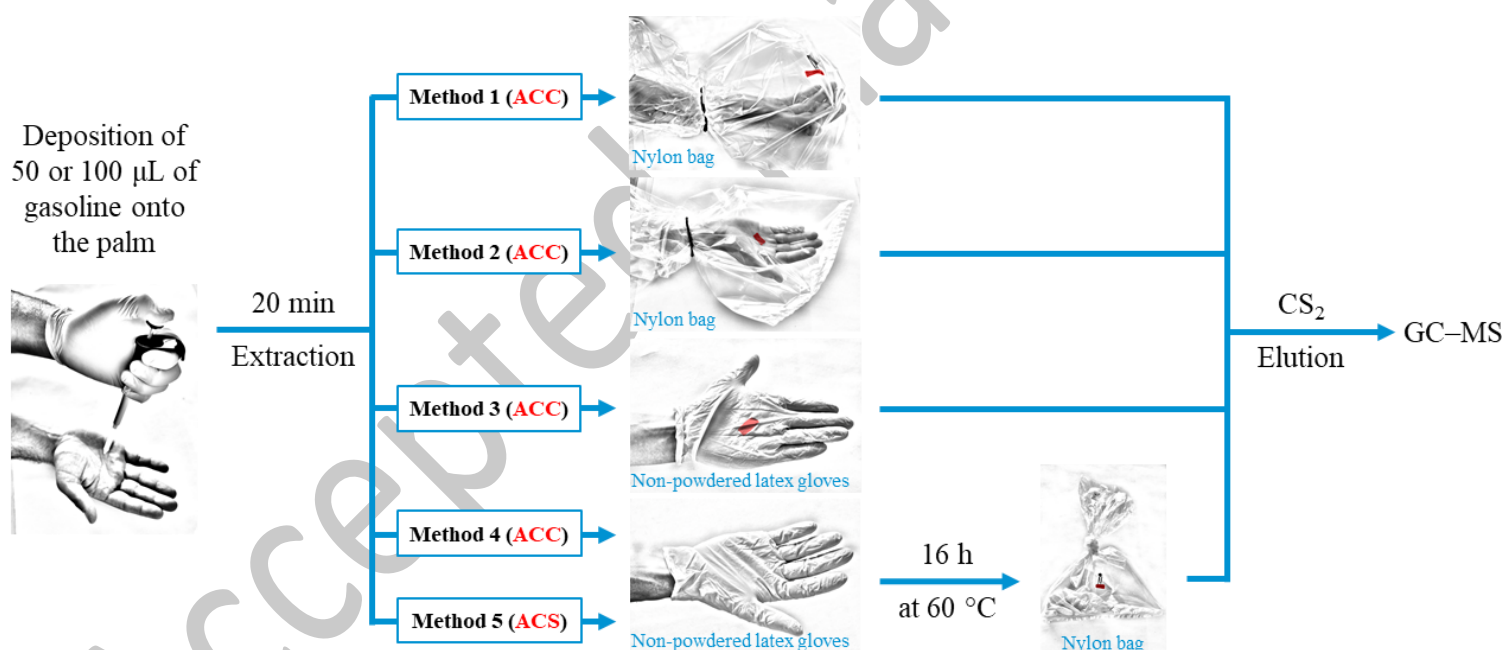# Detection of gasoline on suspects' hands: study of different sampling alternatives

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Graphical abstract

Highlights

- ACC is suitable for the extraction and concentration of ignitable liquid residues

- The sampling ability of ACS and ACC are comparable

- The distance between the skin and the sorbent strongly influences the collection

- The space between the hand and the glove or bag impacts the sampling of gasoline

Abstract

Arsonists may use ignitable liquids to start, accelerate and amplify fires. The sampling of volatiles present on the hands of suspected arsonists is therefore sometimes carried out in the course of the investigation of (possible) deliberate fires. Several collection protocols have been proposed, relying on the concentration of volatiles by the transfer on PVC gloves and further passive headspace extraction with Activated Charcoal Strips (ACS). Previous research findings assessing the use of Activated Carbon Cloth (ACC) – initially developed for the adsorption of gas in military applications – opens the path to new perspectives regarding the extraction and the concentration of ignitable liquid residues in general, and for the sampling on hands in particular. Five alternative methods (four relying on the use of ACC and one on ACS) were considered for the collection of gasoline traces present on the hands and their subsequent analysis by gas chromatography – mass spectrometry.

Gasoline was deposited onto the palms of volunteers to study the differences between the collection method using ACS and those using ACC. For the latter, either the volunteer hands were placed in nylon bags with an ACC on the palm or suspended, or, powder-free latex gloves were used, with an ACC on the palm or in a separate extraction, with the glove in a nylon bag and the ACC suspended.

The results showed that the background contamination was not distinguishable between ACS and ACC and their sampling ability was comparable. The two methods relying on the deposition of ACC directly on the surface of the palm where gasoline was deposited showed significantly higher collection capacity than other methods, provided that the ACC was in direct contact with the contaminated zone. The results showed that three main factors affected the collection of gasoline on the hands: the distance between the skin and the sorbent (ACC or ACS) in case of direct concentration of volatiles on the sorbent, the exposure time, and the space between the hand and the glove or bag (i.e. the headspace volume).

This research opens new perspectives for the sampling of ignitable liquid residues through the use of ACC. It corroborates the perceived potential of ACC for the extraction and concentration of volatile compounds, particularly for fire debris analysis purposes. While the experiments were focused on the collection of gasoline on hands, the results provide valuable information in a more general way for the sampling of fire debris.

Keywords: Ignitable liquid; Extraction; Passive headspace; Activated Carbon Cloth; Activated Charcoal Strip; GC-MS.

1. Introduction

The use of ignitable liquid may occur in the commission of an arson to facilitate the ignition of the fire, accelerate its propagation or intensify the extent of destruction. When the ignitable liquid is spilled on the site, depending on the container to be used, splashes may be transferred to hands or clothing of the perpetrator. The detection of these ignitable liquid traces – or the absence of such traces – may be a relevant clue to consider within the investigation of (possible) deliberate fires. In this perspective, sampling on the hands of suspects or other persons of interest may be worth carrying out during the first few hours after a fire event. One of the methods that has been developed and implemented at the operational level is to place the hands of a person of interest (suspect) in non-powdered latex gloves for 20 minutes [1]. After this period, the gloves are enclosed in a nylon bag and exposed to an Activated Charcoal strip (ACS) by heating the bag to 60 °C for 16 hours. This approach aims at collecting and concentrating remaining traces of ignitable liquids present on hands by transfer on the gloves and further passive headspace extraction [2]. The ACS is then eluted, for instance with carbon disulfide (CS_2), and the extract is analysed by gas chromatography coupled with mass spectrometry (GC-MS) [3].

A few years ago, a material was newly tested to trap vapours of ignitable liquids. Sandercock studied the adsorption capacity of a piece of Activated Carbon Cloth (ACC), a fabric made of activated carbon fibres [4]. He tested the passive adsorption of different ignitable liquids such as gasoline, diesel or kerosene on ACC, and compared it with ACS. The use of ACC by Sandercock is, to our knowledge, the first application of this material in the domain of forensic science. It is already being used in the aerodynamic industry [5] as well as in fabrication of military clothing or items for protection against chemical and biological weapons [6]. It is also used to produce filters for the removal of pollutants such as SO_2, NO_x, or volatile organic compounds (VOCs) in air, or contaminants in wastewater [5, 7]. Another application relying on its large adsorption capacity is the storage of various gases including H_2, CH_4 or CO_2 [8]. Finally, Vermeulen *et al*. used patches made from ACC to assess dermal exposure to benzene and toluene for employees working in shoe manufacturing [9]. Incidentally, these two compounds are also present in some ignitable liquids, mainly gasoline or petroleum distillates [10].

Based on this existing body of research, we therefore conducted a study with a two-fold purpose: first, to assess whether ACC can be beneficially deployed in a method to detect traces of gasoline on the hands; second, to further our understanding of the behaviour of this activated carbon cloth for the collection and concentration of gasoline residues.

Building on existing practices [1, 11-13], five different methods (four relying on the use of ACC and one on ACS) were tested for the collection of gasoline traces present on the hands and further analysis by GC-MS.

2. Materials and methods

2.1. Transfer of gasoline and sampling methods

A sheet of ACC (Flexzorb™ Knitted Cloth, Chemviron®, Belgium) was cut into smaller pieces with dimensions of 10 x 20 mm and stored in a clean box to protect them from contamination.

The experimental transfer of a small amount of gasoline[1] onto the hands of a volunteer was implemented as follows: 50 or 100 μl of gasoline (95 RON, purchased in a BP gas station in the local area) was deposited onto one palm of the volunteer using a Rainin® Pos-DTM MR-1000 positive-displacement pipette (n° 17008580, Mettler-Toledo Rainin, USA). The hands were sampled 2 minutes later using one of the five studied methods[2]:

Method 1 Hands were each placed in a nylon 11 "Fire Debris Bag" (C 64100 "Fire Debris Bag", 300 x 600 mm, 0.04 mm thick, BVDA International, Netherlands) for 20 minutes with a piece of ACC suspended above the palm using a magnet and a paperclip (Figure 1a). The bag was sealed with an elastic around the wrists. The ACC was then removed and eluted.

Method 2 Method 2 was similar to the method 1, but a piece of ACC was directly deposited onto the palm (Figure 1b). A variant of Method 2 (called "**Method 2b**") was also tested by placing the ACC on the back of the hand. The ACC was then removed and eluted.

Method 3 Hands were each inserted in powder-free latex gloves (GPG90, Mutexil, France) for 20 minutes and a piece of ACC was deposited onto the palm (Figure 1c). A variant of Method 3 (called "**Method 3b**") was also tested by placing the ACC on the back of the hand. The ACC was then removed and eluted.

Method 4 Hands were each inserted into powder-free latex gloves (GPG90, Mutexil, France) for 20 minutes (Figure 1d). After this time interval, the gloves were placed into a nylon 11 "Fire Debris Bag" (C 64100 "Fire Debris Bag", 300 x 600 mm, 0.04 mm thick, BVDA International, Netherlands) and a piece of ACC was suspended in each bag, held in place by a magnet and a paperclip (Figure 1e). The bags were placed for 16 hours in an oven heated to 60°C. The ACC was then removed and eluted.

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[1] Gasoline was chosen due to its widespread use in arson [1, 11-13].

[2] The tweezers used for handling the ACC and ACS were previously cleaned with dichloromethane (≥99.9 %, n° 32222, Sigma-Aldrich®, USA) between experiments.

Method 5 Method 5 was similar to the method 4, but using an ACS (Albrayco Technologies® Inc, Cromwell, USA) instead of the ACC (Figures 1d and e). The sampling procedure remained the same as method 4.

For all of these methods, ACC or ACS were eluted for at least 30 minutes with 800 µl of carbon disulfide (≥99.9 %, n° 342270, Sigma Aldrich®, USA) and analysed by GC-MS (see details in Section 2.2).

All methods were tested with an initial transfer of 100 µl of gasoline on hands; methods 2, 3, 4 and 5 were also tested with a smaller volume of gasoline (50 µl).

The five studied methods were also applied on the hands of the volunteer without prior transfer of gasoline, in order to obtain blank samples of the hand matrix. In order to limit the variability of the results, we chose to always use the hands of the same volunteer – a healthy 25-year-old man – which led to certain operational constraints. A maximum of four samples (two on each hand) was collected every day. The same hand was contaminated with gasoline twice during the same time of the day (morning or afternoon). A waiting period of at least ninety minutes was taken between two consecutive experiments, allowing the evaporation of gasoline and limiting the risk of contamination from a previous deposition. Hands were washed twice vigorously with soap before each gasoline transfer.

Each method was replicated between 5 to 8 times to take into account any experimental variability, and the sequence of experiments was randomised to ensure a valid estimate of the repeatability [14].
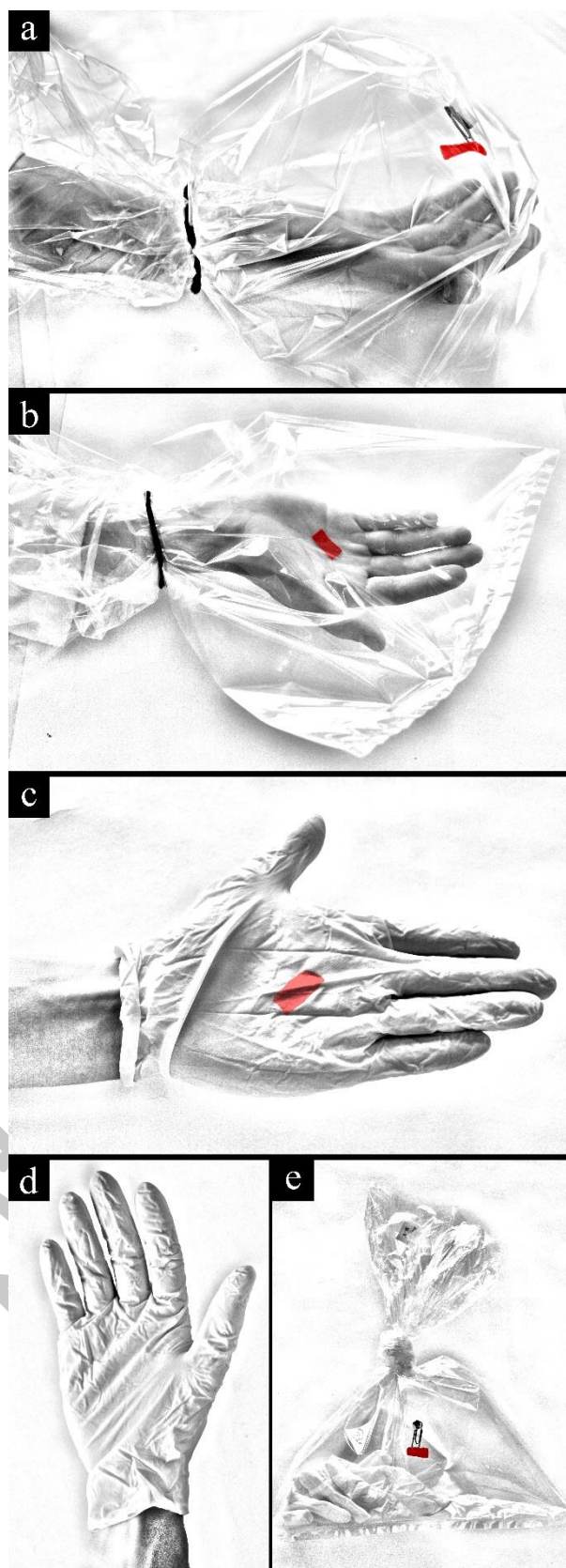
Figure 1 – The different collection methods using ACC or ACS that were studied. a) Hand is placed in nylon bags with a suspended ACC (method 1) or b) on the palm (methods 2 and 2b). c) Powder-free latex gloves were put on hand, with an ACC on the palm/back of the hand (methods 3 and 3b), or without any adsorption device (methods 4 and 5) but with e) a subsequent extraction step into a nylon bag with a suspended ACC (method 4) or ACS (method 5). For visibility reasons, pieces of the ACC or ACS have been recoloured in red on this figure.

2.2. GC-MS analysis

GC-MS analysis was performed on an Agilent 6890N gas chromatograph coupled to an Agilent 5975 mass spectrometer. Injection was performed with an Agilent 7683B Automatic Liquid Sampler, and an Agilent J&W HP-5MS GC capillary column (12 m, 0.2 mm, 0.33 µm) was used for GC separation. The GC-MS fixed parameters are provided in Table 1. Weekly quality control of GC-MS was performed with E-1618-97 Test Mixture (Restek®, USA) to assess the stability of performance of the GC-MS instrumentation during the whole range of experiments. The identification of gasoline in samples was carried out according to ASTM E1618 [3].

Injection parameters	
Injected volume	1.0 µl
Injection mode	Split (25:1)
Injection temperature	250 °C (constant)
Chromatograph parameters	
Carrier gas	Helium (purity: 99.999%)
Carrier gas flow rate	0.8 mL min^{-1} (constant)
Oven temperature program	35 °C for 1.4 min, 10 °C min^{-1} until 250 °C, 250 °C for 9.0 min
Mass spectrometry parameters	
Transfer line temperature	280 °C
Source temperature	230 °C
Quadrupole temperature	150 °C
Ionisation mode	Electron impact (EI)
Electron energy	70 eV
Acquisition mode	Scan
Solvent delay	0.0 min
Full scan mass range scanned	10.0 - 400.0 m/z
Threshold	30
Mass scan rate	3.66 scan s^{-1}

Table 1 – *GC-MS parameters.*

A semi-quantification was performed for sixteen compounds present in gasoline. The semi-quantification method integrated the target ion peak area of the compound of interest based on retention time and the presence of its respective qualifier ions (Table 2). Areas of the targeted compounds were normalised by the 3-ethyltoluene peak area from the E-1618-97 Test Mixture ("normalised peak areas") and performed on a weekly basis prior to analysis, in order to take into account slight variations in the MS response over the duration of the experiments.

N°	Compound	Retention time [min]	Target ion [m/z]	Qualifier ions [m/z]
1	Toluene	2.271	91	92 / 65 / 39
2	Ethylbenzene	3.663	91	106 / 51 / 39
3	m- / p-Xylene	3.795	91	106 / 105 / 77
4	o-Xylene	4.163	91	106 / 105 / 77
5	Propylbenzene	5.164	91	120 / 92 / 65
6	1-Methyl-3-ethylbenzene	5.296	105	120 / 91 / 77
7	1,3,5-Trimethylbenzene	5.410	105	120 / 119 / 77
8	1-Methyl-2-ethylbenzene	5.587	105	120 / 91 / 77
9	1,2,4-Trimethylbenzene	5.806	105	120 / 119 / 77
10	1,2,3-Trimethylbenzene	6.260	105	120 / 119 / 77
11	Indane	6.465	117	118 / 115 / 91
12	1,2,4,5-Tetramethylbenzene	7.738	119	134 / 91 / 133
13	1,2,3,5-Tetramethylbenzene	7.802	119	134 / 91 / 39
14	Naphthalene	8.734	128	127 / 129 / 102
15	2-Methylnaphthalene	10.304	142	141 / 115 / 143
16	1-Methylnaphthalene	10.536	142	141 / 115 / 139

Table 2 – *Retention time, target ions and qualifier ions used for the semi-quantification of sixteen compounds present in gasoline.*

2.3. Evaluation of results

The comparison of the sampling methods was carried out by considering several factors: the presence (or absence) of background compounds originating from the hands, the semi-quantitation of sixteen selected compounds, and the possibility to assess the presence of gasoline according to ASTM E1618. The collection capacity of each method was considered by the sum of normalised peak areas for the 16 target peaks, and represented with boxplots using the Tableau Desktop and R statistical softwares. Moreover, each of these compounds was also examined separately to further assess the efficiency and reproducibility of the methods. Thus, the background contamination and collection capacity were considered to provide an estimate of the sensitivity, selectivity and reproducibility.

3. Results and discussion

3.1. Background noise of the collection supports and blank samples of hands

The background noise of ACC and ACS were studied and tested many times throughout the experimental series (pieces of ACC and ACS were eluted and analysed by GC-MS without prior exposition to hands). No compounds were ever detected on the background ACC or ACS pieces. Similarly, the analysis of blanks of hands allowed to assess the background noise generated by matrix (hands) with each of the sampling methods. No compounds originating from the hands (skin) were detected in the blank samples collected by any of these methods. However, N,N-Dibutylformamide and α-pinene were both present in the extracts from powder-free latex gloves, and the nylon 11 "Fire Debris Bags" were found to contain a small quantity of toluene. N,N-Dibutylformamide and α-pinene were not problematic for our study as they do not interfere with any of the compounds found of gasoline, which is not the case with toluene. But the intensities of toluene chromatographic peaks were negligible in comparison to those in gasoline samples (Figure 2).
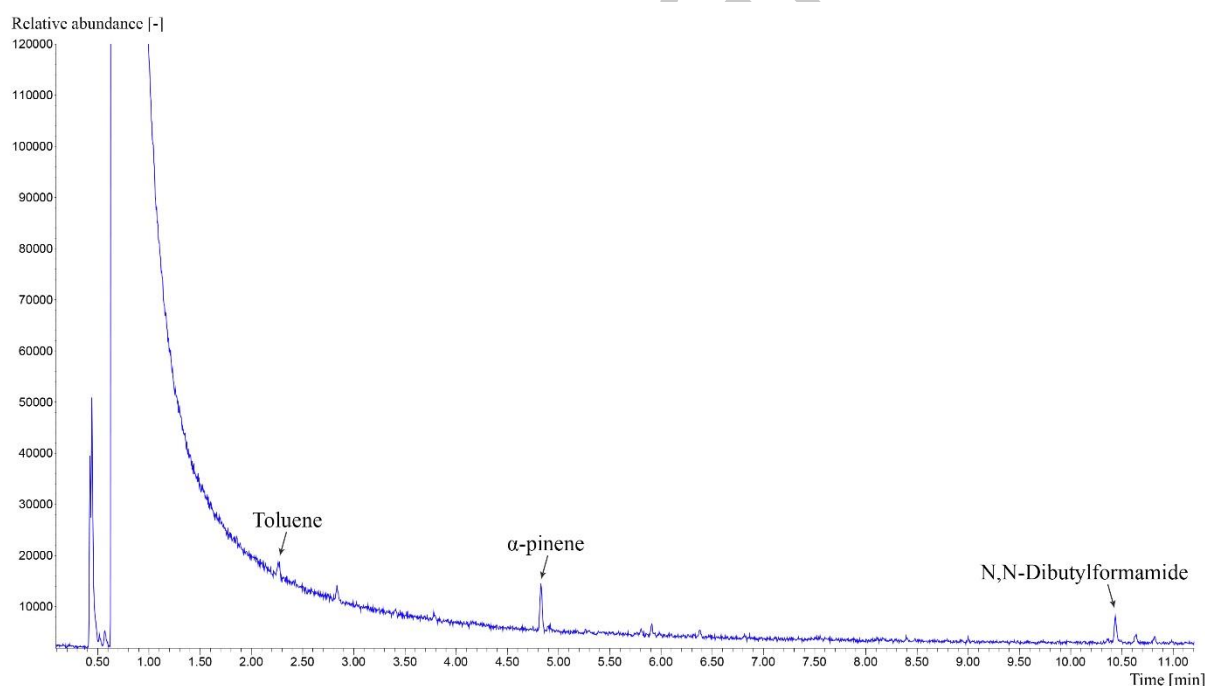

Figure 2 – Representative TIC of a blank sample of powder-free latex gloves and a nylon 11 "Fire Debris Bags" collected by the method 4 (gloves further packed in a nylon bag with an ACC).

3.2. Gasoline identification

All sampling methods tested allowed the detection and identification of the presence of gasoline in accordance with ASTM E1618, except for the method 3b (hands in gloves for 20 min, with an ACC on the

back of the hand). The latter collected only some light molecular weight aromatic hydrocarbons such as toluene or m- and p-xylene. In the end, this method did not meet ASTM criteria for gasoline identification, unlike the others. Figure 3 shows a representative example of a total ion current chromatogram (TIC) obtained for each method with an initial gasoline transfer of 100 µl, and the TIC of a liquid injection of the gasoline used in the experiments. In addition, the presence of gasoline on the hands was detected and identified for the four methods (2, 3, 4 and 5) which were also tested with a smaller volume of deposited gasoline (50 µl).
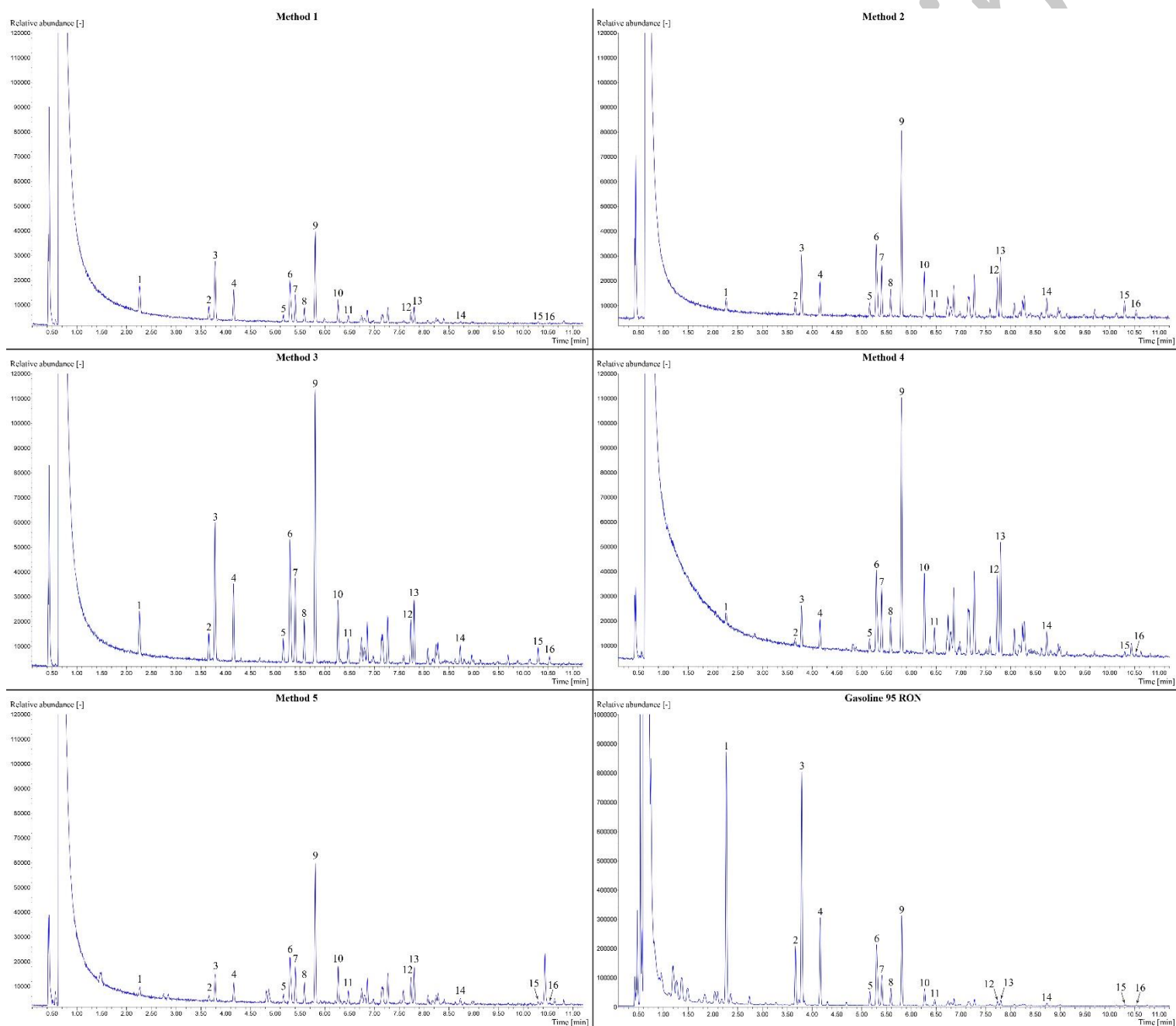

Figure 3 – Representative TIC for each sampling method with an initial transfer of 100 µl of gasoline, and a TIC of the gasoline standard (95 RON) used in the experiments. The sixteen compounds selected are indicated by the number listed in the Table 1. The chromatogram of TIC of the gasoline standard does not have the same scale as the others.

3.3. Collection capacity

The ACC and the ACS were shown to collect the same range of compounds from gasoline deposited on the hands without significant alteration to the relative ratios between structural isomers compared with the neat gasoline (as illustrated by the chromatograms in Figure 3). This observation is in line what Sandercock found in his study on the extraction of various ignitable liquids from sheets of lens cleaning paper [4].

In order to compare the collection capacity of the different methods, we decided to consider the sum of "normalised peak areas" for the 16 target peaks (normalisation to 3-ethyltoluene of the previous quality control), and to represent their variability with boxplots (Figures 4a and 4b respectively with 100 and 50 µl of deposited gasoline). This approach shows that methods 2, 3, 4 and 5 captured and concentrated more volatile compounds than the others (1, 2b and 3b). A further comparison of the methods, considered two-by-two, was carried out by calculating the Student's t-test (95 % confidence interval). Considering both the 50 µl and 100 µl of gasoline transfers, the results indicate that method 3 (gloves with an ACC on the palm) was significantly more efficient in vapour collection than the other methods. Method 2 (nylon bags with an ACC on the palm) was as efficient as method 3 with 100 µl of deposited gasoline, but less efficient in collecting less volatile compounds such as indane or naphthalene with 50 µl of gasoline transfer. Methods 4 (gloves further packed in a nylon bag with an ACC) and 5 (gloves further packed in a nylon bag with an ACS) yielded results that are not significantly different from each other. Similarly, no significant difference was observed between method 1 (nylon bags with a suspended ACC) and method 2b (nylon bags with an ACC on the back of the hand).
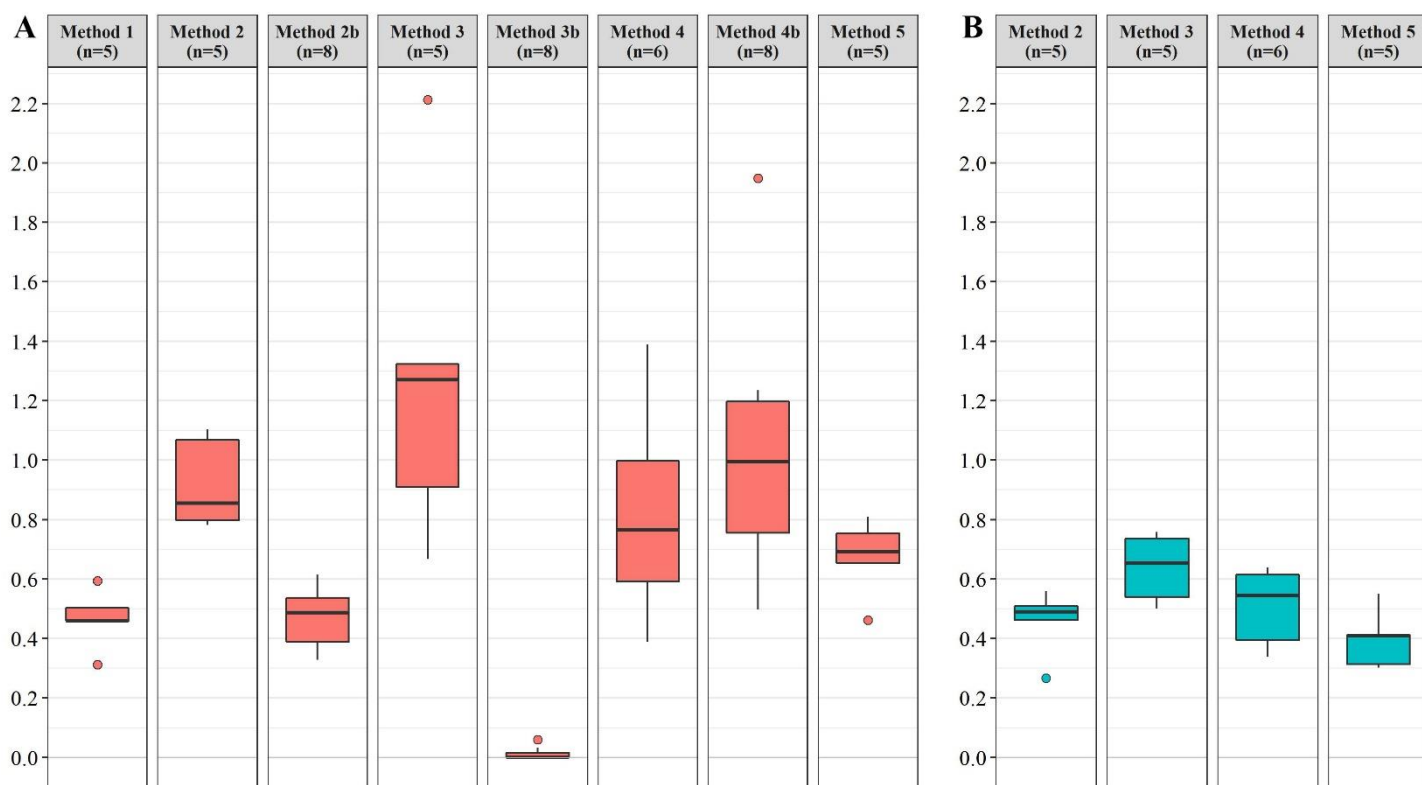
Figure 4 – *Collection capacity of each method with an initial quantity transferred of 100 µl (A) and 50 µl (B) of gasoline illustrated by the sum of normalised chromatographic peak areas of the sixteen compounds selected.*

As shown in Figure 4, method 2 (nylon bags with an ACC on the palm) and method 3 (gloves with an ACC on the palm) were efficient in collecting the 16 targeted compounds of gasoline present on the hands. For both these methods, the sorbent was in (almost) direct contact with the gasoline transferred, since the ACC was placed onto the palm at the location where gasoline was previously deposited. In order to evaluate the influence of the position of the device in relation to the vapour source, variants of methods 2 and 3 (named 2b and 3b) were implemented with the ACC placed onto the back of the hands, with the gasoline still previously transferred onto the palm (Figure 4). No volatile compound was detected using method 3b, (in the gloves), while, all compounds were detected using method 2b (in the nylon bag), but in significantly less amount than method 2.

These results show that two factors seem to have a strong influence on the collection of gasoline vapours: 1) the distance between the location of the source of the vapours and the position of the sorbent device, and 2) the presence of sufficient free space to allow for the diffusion of vapours. A smaller distance between the source of vapours and the sorbent device promotes the transfer of vapours onto the device.

If the sorbent device (ACC in our case) is not in (almost) direct contact with the source of the vapours, the concentration of the vapours on the device is also conditioned by their transport from the source to the

device. The vapours need free space to diffuse and reach the sorbent device. In a previous study, Muller and colleagues made similar observations leading to the recommendation of using a plastic bag rather than gloves to prevent airflow constriction and the formation of air pockets that would hinder the adsorption of vapours on the device [11].

Compared to methods 4 and 5, methods 1 and 2 (with nylon bags) have the advantage of combining vapour collection and enrichment in a single step. On the other hand, methods 4 and 5 are characterised by an easier operational implementation: the first collection stage using gloves can be uncoupled from the second step (vapour concentration with ACC or ACS) and carried out remotely from the laboratory environment, eventually directly in the field.

Methods 4 and 5, rely on the passive adsorption of vapours on the ACC and ACS respectively for 16 hours proved to be slightly more effective at sampling less volatile compounds such as indane, naphthalene or their derivatives (Figure 5) rather than the lighter compounds. This observation is fairly logical in view of the longer exposure time and increased temperature of the device to the vapours, leading to a displacement phenomenon [15]. Conversely, method 1 is more effective in collecting the lighter compounds than the heavier compounds.

On the basis of the encouraging results obtained for a transfer of 100 µl of gasoline on hands, experiments with a quantity half the size (50 µl) were undertaken with methods 2, 3, 4 and 5. The detection and identification of gasoline (according to ASTM E1618) was achieved for each of these methods. Although the sum of "normalised peak areas" of the 16 gasoline compounds selected was significantly lower for each method, we can see in Figure 4 that the response did not decrease as much as the initial amount deposited. Methods 2, 3 and 4 (using ACC) also collected several more compounds than did method 5 using ACS. The only noticeable difference (except the abundance of the response) between the methods relative to the results obtained using 100 µl of gasoline was the predominant amount of toluene – which was in higher quantity in the 50 µl samples relative to other compounds (Figure 6). This change in concentration may be the direct result of the initial quantity of gasoline transferred (no displacement phenomenon taking place).
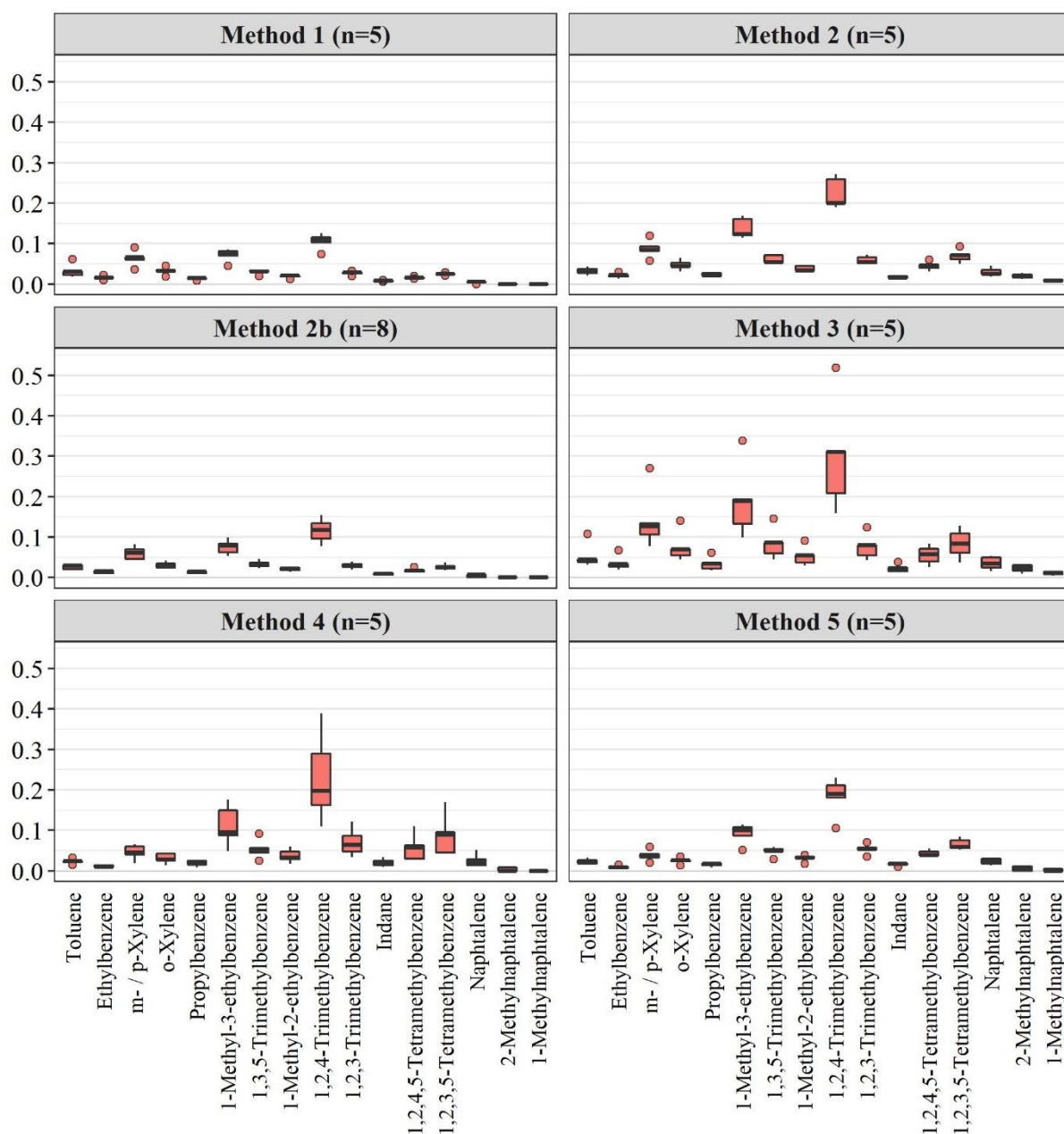
Figure 5 – *Collection capacity of all six methods for samples with 100 µl of transferred gasoline. The figure depicts the normalised chromatographic peak areas of the sixteen compounds selected. The scale is the same for all methods (boxplots).*
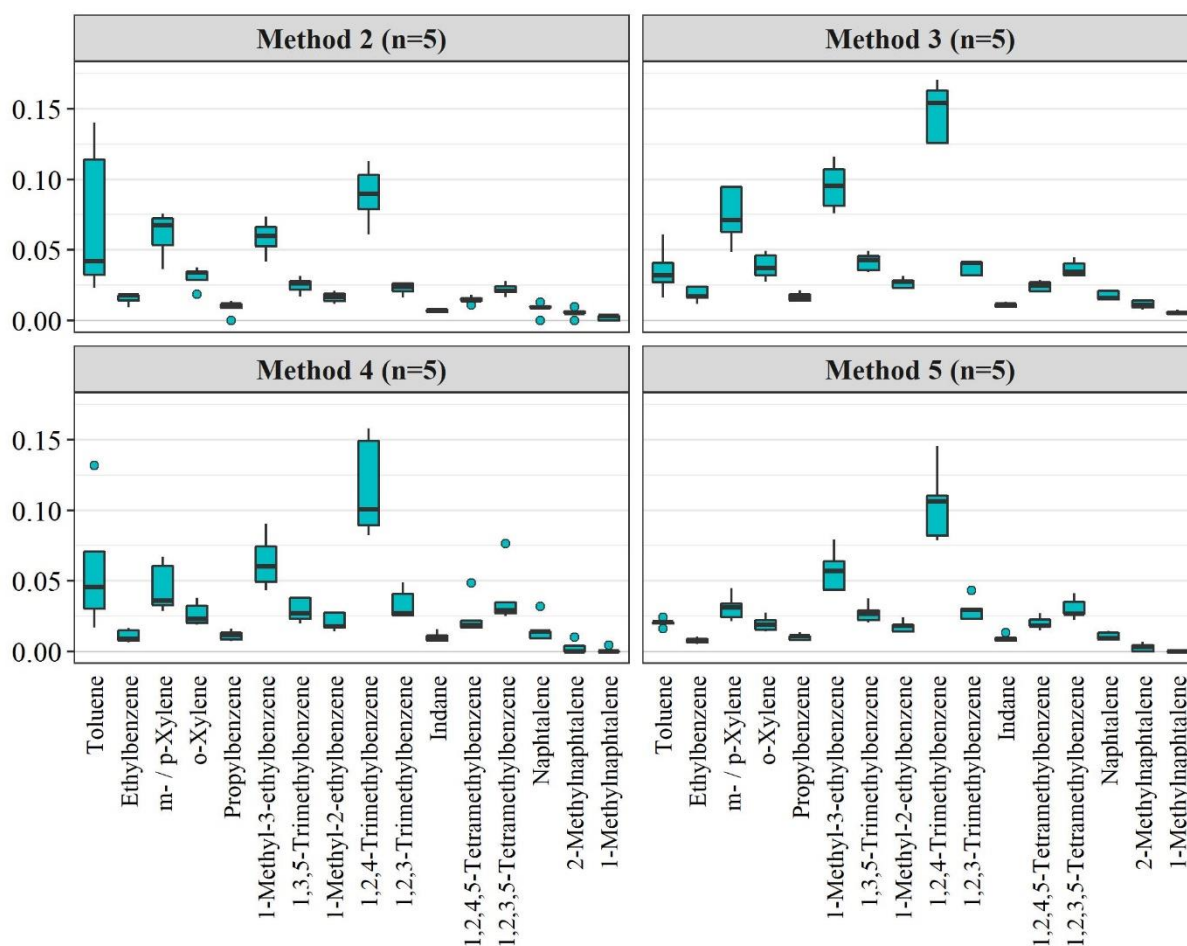
Figure 6 – Collection capacity of the four methods tested for samples with 50 µl of transferred gasoline. The figure depicts the normalised chromatographic peak areas of the sixteen compounds selected. The scale is the same for all methods (boxplots).

It is worth noting that several experimental parameters were not fully controllable, such as the dimensions and the weight of the ACC pieces (cut manually) as well as the volume of air in the nylon bags. The operating procedure was carried out in such a way to minimise the variability induced by these parameters, but they definitely had some influence (depending on the method) on the reproducibility of the results. Figure 4 shows greater variability for the methods using ACC than the one with ACS (method 5), particularly for the experiments with 100 µl of gasoline transferred. This difference in variability may be caused by the variation in size and weight of ACC pieces which were not standardised (as with ACS).

4. Conclusions

This research study examined several alternatives to sample gasoline residues on hands. Five collection methods relying on the use of a sorbent device and on the application of protocols proposed by previous published studies were considered and tested. In addition to evaluating these different alternatives, this study relied mainly on the implementation of activated charcoal cloth (ACC), thus expanding knowledge on the possible application of this still little used device.

In general, ACC has proven to be suitable for collecting gasoline on hands with an extraction efficiency comparable to ACS. Two methods relying on the deposition of ACC directly on the surface of the palm, where gasoline was previously transferred (method 2 with nylon bags and method 3 with gloves) showed significantly higher collection capacities than other methods, provided that the ACC was in direct contact with the location where gasoline had been transferred. These two methods were as efficient as methods 4 and 5 (sampling with gloves, and further extraction with ACC and ACS, respectively) at collecting less volatile compounds such as indane or naphthalene and collected slightly more volatile compounds (ethylbenzene or m- and p-xylene). A small displacement of volatile compound concentrations was observed with methods 4 and 5 due to the re-condensation phase. This study highlighted the influence of two principal factors affecting the collection of gasoline vapours besides the time of exposure: the distance between the skin and the sorbent device (ACC or ACS), and the free space to allow the diffusion of vapours onto the sorbent device.

These results suggest the possibility of using ACC as an alternative to ACS for the sampling of ignitable liquid residues on hands. Further research efforts are, however, still desirable to better delineate the sensitivity and the collection capacity of ACC on a range of ignitable liquids. Future work may also include experiments focusing on the persistence of gasoline on hands, and the possible collection of compounds a few hours after the deposition (using ACC methods); the experimental design of our study relies on a sampling that was always carried out directly after the transfer of gasoline on the hands. The possibility of detecting ignitable liquids residues on hands drops off indeed markedly with time. Thus, the limit of detection becomes an important aspect, as will be the establishment of expected background levels. An improvement in the sensitivity of the analytical procedure could therefore be valuable, e.g. by optimising the headspace extraction and enrichment technique or by modifying selected GC-MS parameters (split ratio).

More generally, this research corroborates the findings by Sandercock's previous study [4], which suggested the use ACC for the sampling of ignitable liquid residues. The present work confirms the perceived potential of ACC for the extraction and concentration of volatile compounds, specifically for fire debris analysis purposes. While the experiments were focussed on the collection of gasoline on hands, the outcomes provide valuable information in a more general way regarding the sampling of fire debris.

Acknowledgements

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