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Instrumental sensing of trace volatiles—a new promising tool for detecting the presence of entrapped or hidden people

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Abstract
There is a growing demand for rapid analytical systems to detect the presence of humans who are either entrapped as a result of a disaster or, in particular, hidden, as in the case of smuggling or trafficking. The trafficking and smuggling of people to Europe have reached epidemic proportions in recent years. This does not only put a major strain on European resources, but puts at risk the health and lives of the people being trafficked or smuggled. In this context, the early detection and interception of smuggled/trafficked people is of particular importance in terms of saving migrants from life-threatening situations. Similarly, the early and rapid location of entrapped people is crucial for urban search and rescue (USaR) operations organized after natural or man-made disasters. Since the duration of entrapment determines the survivability of victims, each novel detecting tool could considerably improve the effectiveness of the rescue operations and hence potentially save lives. Chemical analysis aiming at using a volatile chemical fingerprint typical for the presence of hidden humans has a huge potential to become an extremely powerful technology in this context. Interestingly, until now this approach has received little attention, despite the fact that trained dogs have been used for decades to detect the presence of buried people through scent. In this article we review the current status of using analytical techniques for chemical analysis for search and rescue operations, and discuss the challenges and future directions. As a practical implementation of this idea, we describe a prototype portable device for use in the rapid location of hidden or entrapped people that employs ion mobility spectrometry and a sensor array for the recognition of the chemical signature of the presence of humans.

1. Background

The International Organization for Migration estimates that more than 1 million migrants arrived to Europe by sea in 2015 and around 35 000 by land. However, the EU’s external border force (Frontex) monitoring this phenomenon believes that the actual number of people crossing into Europe in 2015 is nearer to 2 million by taking into account those who are believed to have crossed European boarders undetected. Human trafficking and smuggling and cross-border crime have evolved as a major challenge for the European Union in general and its southern members in particular.

Criminal networks have rapidly reacted to this development and substantially increased their involvement into migrant smuggling. Over the last decade human smuggling and trafficking became two of the fastest growing transnational criminal activities and are thought to be the most lucrative forms of organized crime after the drug trade [1]. Smugglers offer a wide range of services such as transportation, accommodation and fraudulent documents, usually at excessively high prices. Trafficking involves ongoing exploitation and abuse. According to a trafficking report of the Federal Criminal Police Office [2], the number of people picked up during their flight has increased tenfold since 2013. In attempting to reach Europe via the Mediterranean Sea, between 2000 and 2015 more than 30 000 refugees are believed to have drowned or died from hypothermia and starvation. Transport by road can be as dangerous for a refugee. For
example, on the 27th August 2015, on a motorway between Neusiedl and Parndorf, Burgenland, Austria, 71 migrants were found to have suffocated in a refrigerated truck. The detection of smuggled/trafficked persons is therefore of considerable importance not just to protect European borders, but more importantly in terms of the humanitarian aspect in order to rescue people from life-threatening and degrading situations. The early and rapid location of entrapped people is also of utmost importance for urban search and rescue (USaR) operations organized after natural or man-made disasters (e.g. earthquakes, tropical storms, or terrorist attacks). These events cause not only many deaths, but also numerous traumatic injuries and entrapment of survivors in collapsed buildings. While about 50% of survivors are found and rescued quickly by bystanders, the remaining ones are subjected to prolonged entrapment under complex debris [3]. The survivability of victims depends on several factors; however, it is primarily related to the duration of entrapment [4]. Recent studies have demonstrated that the vast majority of survivors (80%) are found and extricated during the first 48 h of entrapment [3]. After this period (called frequently the ‘Golden 48 h’) the survival rate drops rapidly and after more than 5 days from the time of entrapment only victims entrapped under favourable conditions (e.g. having access to water and/or food) will survive.

Currently, the presence of trapped humans is detected through the use of search and rescue (SAR) dogs. These are commonly recognized as a golden standard in this context [5]. Search dogs have excellent scenting and hearing skills, are able to search relatively large areas in a short period of time and can work in areas that are deemed unsafe or inaccessible to human rescuers. However, they exhibit a number of limitations [6, 7]. Their working time is relatively short, being restricted typically to approximately 30 min (with a subsequent necessary break of 2 h) and their training is time-consuming and expensive. Moreover, their work can be affected by their personality and physical needs, they respond poorly if stressed, bored or frustrated and can easily be injured in highly toxic and harsh disaster environments [6–8]. These limitations have resulted in a need for novel analytical tools, which could complement or even replace SAR dogs during USaR operations. A number of technical tools have been applied in this context. These embrace a number of technologies, including e.g. fibre optic cameras (borescopes), acoustic probes (aiming at voices or detecting heartbeats), thermal cameras, and sonars [9]. In addition to these, carbon dioxide (CO₂) sensing has been employed to detect stowaways hidden inside shipping containers or trucks [10]. These instruments undoubtedly improve the effectiveness of locating victims; nevertheless, the role of canines in USaR operations remains still invincible.

The fact that SAR dogs can detect survivors in highly contaminated disaster sites implies that there is a human-specific chemical signature in the surroundings of entrapped or hidden people and that the analysis of this signature could be used as a valuable detection tool. However, an analytical approach has received little attention and is limited to the aforementioned CO₂ sensing. This is surprising given that volatile chemical compounds are often the final products of vital metabolic pathways occurring in the human organism and hence could therefore serve as signs-of-life in the context of rescue operations. Indeed, there is growing evidence provided by a number of recent studies suggesting that some constituents of the human scent could be employed for this purpose and thereby considerably improve the effectiveness of rescue operations [11–13]. Apart from the detection of victims, chemical analysis of the surroundings could provide the rescuers with the capability to recognize exposures to potentially toxic agents which can be present at disaster sites [14].

2. Potential markers of human presence

Volatile species forming the human scent during entrapment can stem from compounds present in human breath or emitted from urine, faeces, blood, sweat and skin. Their sources can be classified into continuous and temporal ones. The former group, embracing breath and skin emanations, is particularly important in the context of human detection, as it offers a long-lasting emission of potential markers of human presence. Moreover, breath holds a distinguished status since the breath-borne volatile species could be used to differentiate between living and dead victims. Temporal sources such as urine or blood have a more transient contribution to human scent; nevertheless, they cannot be neglected. The occurrence of volatiles from these sources is difficult to predict; however, it is reasonable to assume that in the context of humans entrapped in collapsed buildings that the emission of blood-borne species will appear at the early stage of entrapment as a result of injuries induced by the disaster. Furthermore, urine- and blood-borne compounds are expected to strengthen the location signal provided by breath markers of human presence due to the physiological dependencies between these fluids. However, these sources should be considered as limited reservoirs of species tending to dry out.

The emission rates of volatiles from the aforementioned sources depend on the physiological and medical status of the victim (e.g. injuries, dehydration, shock, diet, history of environmental exposure, drug intake, etc.), conditions in the entrapment scene (e.g. restricted space volume, temperature, humidity, and oxygen content) and the time of entrapment [13]. Although the origin of many volatiles emitted by the human body remains ambiguous, several sources could explain their occurrence. These include (i) metabolic production related to the physiological processes in the body (both normal and abnormal), (ii) oxidative stress (e.g. on skin surface), (iii) metabolism
of human microflora, (iv) environmental/medical/industrial exposure, and (v) diet and its metabolites. The origin, production and emission rates of these species are still far from being completely understood; however, the recent rapid advances in metabolomics may help resolve this problem in the near future.

An ideal marker of human presence should be omnipresent in the human volatolome, distinguishable from background levels, relatively non-reactive, continuously emitted by the human body and present in the proximity of an entrapped person at levels detectable by portable field analysers.

A number of studies illustrated the capabilities of different analytical techniques (gas chromatography-mass spectrometry (GC-MS), proton transfer reaction mass spectrometry (PTR-MS), chemical sensors, or ion mobility spectrometry (IMS)) for identifying potential markers of human presence in breath, skin emanations, urine, or blood [11, 12, 15–23]. These reports provided data that assisted in the formation of a preliminary database of volatile compounds associated with human scent, and hence have the potential of being used as markers of human presence [13]. Talking into consideration only reliably identified and quantified species, 47 chemical compounds have been pre-selected using skin emission and exhaled breath data. Within this set of species the most numerous chemical classes are aldehydes (23%) and hydrocarbons (21%) followed by ketones (13%) and inorganic compounds (9%). The quantitative data on these species in breath and skin emanations have helped to estimate their approximate emission rates from humans as paramount factors determining their concentrations in the neighbourhood of a human. Excluding CO₂, the mean fluxes of volatiles of interest were spread over several orders of magnitude and ranged from 0.03 to 600 nmol × min⁻¹ × person⁻¹. These can be used to determine concentrations as a function of distance from a human. For example, at a distance of 3 m from an entrapped person and for a debris-to-air ratio of 3:1 these emission rates may generate concentration levels in confined spaces of collapsed buildings ranging from 0.1 ppb to 1 ppm [13]. Although, the aforementioned database should be considered as initial and the predicted concentrations in void spaces as tentative (to be complemented and verified under field conditions) they can be used to support the selection of an optimal field analytical technique for the real-time detection of trapped humans.

The pre-selected markers were classified into three subsets of different potential detection. The major classification discriminants were (i) their emission rates, (ii) the capability of their detection by portable, real-time analytical instruments and (iii) their background levels in an urban environment. Following this classification 11 metabolites (CO₂, ammonia, acetone, 6-methyl-5-hepten-2-one, isoprene, n-propanal, n-hexanal, n-heptanal, n-octanal, n-nonanal, and acetaldehyde) are classified as class A, comprising predominantly systemic species exhibiting high emission rates from a human body, which are distinguishable from urban background levels using currently available portable field analysers [13].

3. Analytical instrumentation for field detection of potential markers of human presence

The real-time location of humans via chemical analysis imposes a number of limitations on potential field analytical techniques, where simple-in-use (‘yes/no’ response), rapid, hand-held, low energy and simultaneously sensitive and selective screening instruments are required. Two techniques arguably have the greatest potential in this context, namely IMS and electronic sensors.

IMS separates volatiles on the basis of differences in the drift speed of product ions in an inert buffer gas under the influence of an electric field [24]. Recent rapid advances in IMS have resulted in the development of numerous sub-techniques exploiting different forms of the electric field (e.g. linear drift tube IMS, aspiration IMS (AIMS), or field-asymmetric IMS (FAIMS)), or combining IMS with other techniques such as, e.g. GC (GC-IMS). To date IMS has mainly been used in homeland security applications predominantly for the detection of warfare agents and explosives, but also for illicit drugs. However, this technology has also proven itself useful in several civilian applications [25–30]. IMS instruments are portable, robust, measure rapidly, have low energy consumption, and are very sensitive. Without pre-separation or mass spectrometric measurements, selectivity is an issue. Nevertheless, IMS is capable of near-real-time detection and discrimination of numerous markers of human presence (e.g. ammonia, ketones) at low ppb levels without sample pre-processing. Moreover, the expertise and know-how gained within the current field applications of this technique could be transferred into the safety and security sector.

Recent progress in electronic sensor technology has resulted in the development of devices known as electronic noses (e-noses) [31]. The e-nose technology is already frequently applied in the food and beverage industry for product classification and for quality control. Generally data handling methods such as principal component analysis, hierarchical cluster analysis, and linear discriminant analysis are used to decrease the dimension of the acquired odour data, while machine learning and decision support systems are generally used during classification. [32] With more research and development, they are likely to become a field detecting tool, since they are small, simple to use, and inexpensive. These instruments are matrices of different sensors capable of detecting and discriminating a wide diversity of chemical species.
The capabilities of e-noses stem from the variety of sensors available for the selection of sensor arrays (e.g. chemiresistors, metal oxide sensors, mechanical oscillators, colorimetric sensors etc) and the abilities of manufacturers to produce customized, low-cost, and multi-use devices for particular applications [31, 33–35]. If successful, sensor matrices may revolutionize the chemical analysis of human scent. Once built into robust and small instruments, e.g. in borescopes, they could screen the interiors of confined spaces for volatile chemical signs-of-life.

4. Challenges and future directions for humans location based on chemical analysis

In the context of rescue operations, the chemical detection of volatile markers can be considered as a very promising approach, but it is still in its infancy. Owing to the specific nature of this field, ethical and methodological requirements place restrictions on the studies of markers associated with human presence. These pose considerable challenges and problems. Therefore, before the chemical analysis will be accepted by rescue teams and introduced as a routine location tool during USaR operations a number of points (i)–(iv) have to be addressed.

(i) Potential markers of human presence have to date been selected on the basis of studies involving humans under normal conditions and thereby should be considered as tentative. The initial selection of potential markers relies mainly on the existing literature data on volatile organic compounds (VOCs) in breath or from skin emanations coming from humans under normal conditions. Here, the omnipresence of VOCs and the level of their emission from human body can be recognized as the first selection criteria. Since the quantitative data (emission rates) for skin and, in many cases, breath volatiles are relatively sparse the evaluation of the real VOCs fluxes from human body may suffer from the shortage of reliable data [13]. Here, the whole body emission data can be considered as the most valuable [12, 20]. Thus, the future validation of markers under conditions mimicking the environment of confined spaces is highly desirable. For instance, this could embrace the monitoring of VOCs in shipping containers containing human subject(s).

(ii) The evolution of the human-specific chemical fingerprint during prolonged stay in confined spaces is poorly known. Prolonged entrapment results in notable changes in physiology and biochemistry owing to numerous processes resulting from systemic complications and a number of neuroendocrine, metabolic, and physical responses [4, 36]. These include emotional stress, dehydration, starvation, or asphyxiation. Little is known how these conditions affect the production and emission of volatiles from the human body in general and markers of human presence in particular. Thus, the emission of some markers abundant under normal conditions may be considerably changed following prolonged confinement. For example, the entrapment conditions cut off the subject from the factors inducing oxidative stress on the skin surface resulting from exposure to UV radiation or O₃. Consequently, it can be expected that the skin emission of the oxidative stress related species will be reduced shortly after entrapment [13]. Again ethical restrictions limit laboratory-based studies to investigate this. The available literature reports are therefore sparse and provide data obtained for a relatively short periods of entrapment, too short to observe changes in the human metabolism [12, 20].

(iii) The interactions of human-related chemical fingerprint with the entrapment/location environment have not been investigated in sufficient depth. Once emitted, volatiles which form the human scent are spread throughout the void spaces, interact with surrounding materials, and mix with contaminants present in the environment [13]. Additional factors affecting the VOCs' concentrations in the void spaces are temperature and especially high humidity, inducing condensation and formation of water films promoting wet chemistry. All of these factors and confounders may considerably affect the concentrations of the human-specific volatiles and make their identification and detection a challenging task. This results in the usefulness of some promising potential markers to be considerably limited owing to the variable and unpredictable levels of background VOCs, or through the complexity of the location sites. Moreover, the matrix and environmental differences between sniffing for buried people and sniffing for smuggled people have to be taken into consideration. The latter may result in a different set of markers to be used for these scenarios. Given that these problems have so far received little attention, the successful application of chemical analysis in rescue operations requires thorough investigations of the aforementioned issues. Certainly the detection of smuggled persons requires the determination of chemical patterns representative for shipping containers or trucks containing different cargos and having different conditions (e.g. temperature). Such a library of patterns would help to validate potential markers against the possibility of their detection in the contaminated environment.
of shipping containers and assist in the selection of optimal field analytical instruments capable of detecting humans in harsh and contaminated disaster environments. Moreover, a major focus should lie on the investigations of interactions of volatiles of interest with containers’ materials and other adsorbents such as clothing, dust, or soil. A conjunction of canine olfaction with instrumental analysis could be here an interesting approach accelerating the selection and validation of human occupancy markers [6, 7].

(iv) Potential field analytical instruments have to be optimized toward the detection of a human-related chemical fingerprint. The success of chemical analysis for human detection primarily depends on the availability of analytical technologies to provide rapid, continuous, and field detection of volatile signs-of-life. Here, the selectivity, sensitivity (e.g. the limit of detection) and the ability of the technique to distinguish markers or a fingerprint of interest from the background play a fundamental role. However, other features such as portability (miniaturization), low energy consumption, or ability to work in harsh, contaminated, and even toxic environments cannot be neglected. Thus, the future optimization of field techniques for the detection of biomarkers of interest could considerably improve the effectiveness of a rescue operation. However, a key prerequisite is the identification of robust and reliable markers of human presence, which would allow manufacturers to produce customized, low-cost, and multi-use devices for targeted monitoring. Moreover, such devices will have to be developed to recognize human-related fingerprint (or markers) under different environmental conditions. This requires the creation of a large database of chemical patterns typical for different compositions of urban air, indoor air inside transport containers containing different goods and materials, fragrances, headspaces of bio-waste, building materials and many others in order to identify, understand and hence minimize the effects of confounding factors. The complexity and unpredictability of the entrapment environment render these efforts an extremely challenging task.

5. Prototype of portable locator employing chemical analysis being developed by the Breath Research Institute at the University of Innsbruck

5.1. A prototype design
A portable device is being developed by us which combines an infrared (IR) camera and different gas sensors, among them an aspiratory IMS, and metal oxide semiconductor and field-effect transistor sensors [37]. These are integrated in a transport box together with a lithium–iron phosphate battery, and a control unit for the sensors, the camera and communication module (figure 1). The housing is water resistant and can be combined with a backpack carry system. It is planned that the sensor system will be controlled via WLAN by a smart phone and/or tablet.

The IR camera (Tamarisk 320 AF40, DRS Technologies, Inc., Dallas, Texas, USA) is located at the end of an extendable sampling tube. Different IR cameras were tested in order to determine which one is the most suitable. Weight and size, response time and spectral resolution of these cameras were important factors taken into account when making the final selection. The gas sample is collected from the end of the extendible rod through a special sampling system comprising of an inert tubing, a dust separator and a pump. The pump is used to drive air into the gas sensor system. For sealed containers with only one plug hole for condensed water, the sampling stick can be exchanged with a flexible thin tubing, or borescope, which enables gas sampling without the requirement of opening the container doors.

The AIMS consists of an ionization region and a separation region. During analysis the air sample is pumped continuously into the IMS cell, where the air molecules become ionized by the 241Am-radioactive source. Ion–molecule reactions with the trace VOCs produce signature product ions which are separated according to their mobility and then detected as a current pulse. To operate in either positive or negative ion mode, the device can be rapidly switched between the two polarities. A histogram of ion current at each electrode is used to represent a spectrum. The histogram pattern produced provides the fingerprint, without the identification of the individual compounds. Figure 1(C) shows a histogram representing a typical ‘breath-fingerprint’ measured by the AIMS.

As discussed earlier, the gas composition of human emission samples and also the surrounding air is extremely complex. Thus the unambiguous assignment of VOCs that are coming from humans is challenging. Hence the proposal is to work with chemical fingerprints rather than to try to identify individual volatiles. For this a calibration system still needs to be developed to regularly monitor the accuracy and sensitivity of the instrument.

The basic mechanism of the aldehyde sensor is the oxidation of aldehydes to the corresponding acids. The oxygen consumption is measured electrochemically. The selectivity for aldehydes with a low number of carbon atoms (1–10) is possible by the use of a specific porous polytetrafluoroethylene membrane, the composition of the working electrode and the electrolyte, and the applied voltage across the electrode. The aldehydes are oxidized at the anode. The electrons produced by the oxidation of aldehydes are consumed at the cathode. According to test measurements by Obermeier et al acetaldehyde and a mixture
of different small-molecule aldehydes (C1–C10) can be detected with a good sensitivity down to the lower ng l\(^{-1}\) range [37].

5.2. Preliminary results
In order to ‘train’ the sensor system to recognize diverse odours, different human fluids, such as urine, breath, sweat, and blood have been measured and characterized with the sensor system and lab-based analytical devices (e.g. with different mass spectrometers). Moreover, pre-selected air samples representative for the ambient air, indoor and outdoor air at different locations, head-space air of various packaging materials and food products have also been analysed. A detailed description of the applied AIMS unit and sampling protocol is given elsewhere [38]. Samples were located in an in-house made Teflon chamber (3 l) equipped with gas inlet and outlet. During analysis the gas outlet was connected to the sensor device and air was drawn through the chamber by the use of an internal pump of the sensor system, where the characteristic chemical fingerprints were determined. For breath sampling, subjects exhaled directly into the sampling tube of the device from a distance of 1–2 cm, whereas skin emanation sampling was accomplished by placing the inlet tube on the skin (parallel to the surface). Altogether 44 human samples and 44 non-human samples were analysed.

With help of the ‘random forest’ method, human and non-human compounds were easily distinguished. Sensitivity and specificity were selected as statistical measures of the performance of a binary classification test. The sensitivity and specificity were both around 0.8. The area under curve of a receiver operating characteristic (ROC) curve was determined as 0.8998 (figure 2). The random forest method performs by using leave-one-out cross-validation, ensuring a better quality for sensitivity and specificity by guaranteeing the difference of the training set and sample set. Figure 2 shows an exemplary case for a ROC curve calculated from the samples collected mainly under laboratory conditions, but the general shape is expected to be similar for field applications. The ‘working point’ can be selected according to the required specificity in the field during a search scenario.

A ‘fingerprint database’ is currently being developed and complemented for samples taken under different field scenarios, involving volunteers of different ages. It is planned that specific field testing will be performed in cooperation with the Austrian Ministry of Defence and Sports and Austrian Ministry of Interior.
Joint Operational Office Vienna against Human Smuggling and Human Trafficking).

6. Conclusion

In the context of an escalation of human smuggling and trafficking, the demands for rapid analytical systems for non-intrusive inspections of cargoes and the detection of people hiding inside cargo containers are expected to increase. The analysis of VOCs aiding in the detection and location of people has been considered in this paper as a promising approach, opening exciting research possibilities in the area of security and surveillance. If successful, it may revolutionize rescue operations and save thousands of lives. The preliminary studies presented here are very promising and it is expected that advances in analytical chemistry will resolve the several technical problems and limitations highlighted in this paper. The major limitations for progress are the ethical challenges as well as the complexity and unpredictability of the entrapment environment, rendering the development of a portable battery operated detector of human presence a challenging task. Nevertheless, it can be anticipated that following a phase of extensive scientific and user-field testing trials, a new generation of devices capable of real-time detection of entrapped or hidden people would become a powerful tool in the arsenal of rescuers as well as immigration control authorities.

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