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Evaluation of an innovative proposal for the integration of chemical sensors with SPME fibers on UAVs, for forensic, military, and civil applications

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Abstract This work proposes the innovative integration of chemical sensors, specifically photoionization detectors (PID) and metal-oxide (MOX) sensors, with solid-phase microextraction (SPME) fibers on unmanned aerial vehicles (UAVs). This combination, proposed for the first time, enables rapid detection, sampling, and identification of volatile compounds via gas chromatography–mass spectrometry (GC–MS). PID and MOX sensors provide real-time data on a wide range of compounds, while SPME fibers enhance sensitivity and allow post-flight analysis of absorbed substances. The system detects chemical warfare agents (CWAs), volatile organic compounds (VOCs), and toxic industrial chemicals (TICs), supporting remote and early-stage event detection. A low-cost laboratory setup was developed to test sensors and SPME under controlled conditions, optimizing configurations and titration curves prior to UAV deployment. This work represents a significant advancement in environmental monitoring and emergency response, offering safe, efficient, and versatile detection capabilities.

1 Introduction

In an era characterized by evolving security challenges and rapid technological advancements, the integration of innovative approaches has become essential for effective risk management and threat detection. This study is a journey that explores the potential of hyphenating advanced chemical sensing technologies with the capabilities of Unmanned Aerial Vehicles (UAVs) to address critical concerns in the field of forensic analysis, military operations, and civil protection. By harnessing the power of Photoionization Detectors (PIDs) and Metal-Oxide (MOX) sensors in tandem with Solid-Phase Microextraction (SPME) fibers, this work aims to explore the possibilities of using these three technologies together for rapid and accurate compound identification in difficult operational fields. A non-exhaustive list of operational scenarios can include: industrial accidents, arsons, terrorist attacks, and leakage of chemicals during transport. Furthermore, the utilization of Gas Chromatography–Mass Spectrometry (GC–MS) serves as a tool in identifying the compounds, thereby allowing a comprehensive and integrated approach not only to Chemical Biological Radiological Nuclear explosive (CBRN) threat detection and analysis, but also for forensics and civil purposes where detecting and sampling unknown and potentially toxic chemicals must be performed quickly and remotely. As we explore the characteristics of chemical sensors, SPME, UAVs, and the experimental setup, the subsequent sections of this paper explain the potential implications and applications of this integrated framework, offering insights that span across sectors and domains.

Indeed, the next chapters are dedicated to the description of the peculiarities of the three components of this work: the chemical sensors, with a specific attention on the PID and MOX that are the most useful in the field of gas detection; then, the SPME, its use, and the different field of applications; then, the UAV's and the Italian legislation with a glimpse on the use of the drones for the police forces. Last, the description of the cheap equipment that is proposed to test in steady and controlled conditions the response of the PID and MOX sensors and the SPME to the exposure of chemicals. This experimental work suggests a cheap but precise equipment, to test in a laboratory-controlled condition, the response to the same chemicals by different chemical sensors and to the SPME, allowing to determine the LOD and LOQ of the target compounds.

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Table 1 Advantages and drawbacks of PID sensors

Advantages	Drawbacks
High sensitivity (up to ppb)	Limited to ionizable gases
Fast response and real-time monitoring	Can detect different volatiles but they do not distinguish them
Sensitive to several classes of compounds	They are prone to interference from humidity and temperature
Non-destructive measurements	Require a calibration depending on the specific application and environment
Broad range of detectable compounds	Potential for sensor saturation
Portable and easy to use	Cross sensitivity to other compounds

1.1 Chemical sensors

In the field of environmental monitoring and dangerous chemicals detection, using the combination of the chemical sensing technologies has shown to be a good solution to address the issue of monitoring the VOCs or TICs or CBRNe-related substances or compounds of forensic interest. Chemical sensors have proven to be effective in this field, being more and more miniaturized, lightweight, very sensitive, and with a very quick response. Among all the chemical sensors available, two distinctive types, Photoionization Detectors (PID) and Metal-Oxide Sensors (MOX), have acquired great attention for their different applications and optimal capabilities of low cost and effectiveness, also mounted on drones. These sensors offer rapid, accurate, and real-time detection of a large quantity of compounds.

The Photoionization Detector (PID) uses the principle of the photoionization effect. Employing the effect of the ultraviolet (UV) light to ionize a diverse array of Volatile Organic Compounds (VOCs) and inorganic gases, PIDs can detect with a remarkable sensitivity a great number of hazardous substances and gases in a wide range of concentration. The ions and electrons generated in the ionization process create a measurable current, allowing the determination of gas concentration.

Metal-Oxide Sensors (MOX) use conductivity variations induced by gas oxidation of a surface. A semiconducting layer in the MOX sensors modify its electrical resistance, while oxidizing or reducing gases (or vapors) came in contact with the sensor. Their sensibility ranges from the ppb to the ppm. MOX sensors have then the capabilities and the characteristic that make them another tool that can be used in combination with the UAVs.

Other chemical sensors have been produced and are currently being used and developed as portable solutions, but among them, now, PID and MOX sensors have the most attractive advantages to be mounted and coupled with the drones. In the following paragraphs, the principles of functioning, advantages, and drawbacks of these sensors are clarified, to show their potential and limitation in chemicals identification.

1.1.1 PID sensors

A Photoionization Detector (PID) sensor is a device that can be used to detect a wide range of volatile organic compounds (VOCs) and certain inorganic gases. PID could be low cost, miniaturized and lightweight[1]. Its principle of operation is the photoionization effect, induced on the compound of interest by a high-energy UV light. The generated positive ions and free electrons are attracted to the oppositely charged electrodes. The small electric current generated by the movement of these charges is proportional to the concentration of the ionized molecules in the chamber. The more molecules are charged positively, the more current is generated.

The sensitivity of these apparatuses is very high; the molecules can be detected at very low concentration; the sensitivity depends on the energy of the lamp and of the ionization potential of the molecules: the energy of the UV lamp must be higher than the potential of the molecule; otherwise, the molecule does not go into photoionization and the signal is not produced.

High sensitivity, quick response time, and portability make these sensors suitable for VOCs, CWAs, and TICs rapid response; nevertheless, they lack specificity, the energy ionization of the target molecules must be consistent with the ionization energy of the chosen lamp, and they are prone to interference from humidity. For example, the ionization energy for CWAs ranges from 9.68 eV of Mustard gas to 13.6 of HCN (Tables 1 and 2).

In the following table, the major advantages and drawbacks of PID sensors are listed.

1.1.2 MOX sensors

The principal use of the Metal-Oxide Semiconductor (MOX) sensors is to detect gases and volatile organic compounds (VOCs) in the environment. These sensors operate basing on the principle of conductivity changes in the presence of target gas molecules.

The change in conductivity is measured by the electrodes and converted into an electrical signal proportional to the gas concentration. This signal can be processed and displayed for monitoring purposes. There are various types of MOX sensors, with different metal oxides that have a specific gas target and application.

In 2, the major advantages and drawbacks of MOX sensors are listed.

Table 2 Advantages and drawbacks of MOX sensors [2]

Advantages	Drawbacks
Fast response and recovery time	High operating temperatures
Small size	Higher power consumption respect to other sensors of other materials that are no metal oxides
Inexpensive to produce, thin film of metal oxide	Influenced by environmental conditions, such as dust, moisture, and temperature
Lower power consumption than thick film sensors	Require a calibration depending on the specific application and environment
Can be integrated directly into measurement circuit	Can detect different volatiles but they do not distinguish them
Highly sensitive	

1.2 SPME

The SPME (solid-phase microextraction technique) was invented by prof. Pawliszyn, from the University of Waterloo (Ontario, Canada). At the beginning, the SPME was a chemically modified fused silica fiber. Since then, the technique has evolved with several substrate and coating for the fiber and has gained a great success in many application fields.

The fiber itself is contained into a hollow needle, that in the GC acts as a septum piercing needle; the fiber can be exposed from the needle to sample and then retracted; for the analysis, the needle is pushed through the septum and the fiber exposed into the liner for desorbing the adsorbed species, that are forced into the GC column and analyzed.

The fiber could sample the analytes from a liquid, by direct immersion, or in the case of volatiles sampling, the fiber is exposed in a headspace vial/container or in the air for environmental sampling. In direct immersion or volatile sampling, the phases are the same, the only difference is that in direct immersion, the fiber is immersed into a liquid, while in headspace sampling, the fiber is exposed to the volatiles.

The fibers have essentially two shapes: the classic SPME and the SPME arrow. The difference is that the arrow SPME is rugged, and the arrow-shaped end of the fiber closes the needle preventing from exposing it to unwanted substances before the use. Moreover, SPME arrow has better capacity and extraction efficiency compared to a classical SPME.

Before the use, the fiber must be conditioned at a temperature and for the time that is indicated by the producer.

Several phases for the SPME have been developed; each phase has different characteristics, due to the thickness of the fiber and/or the material of which they are made; the phases differ for the affinities with compounds of different MW and/or polarity.

To choose the appropriate SPME phase, some criteria must be considered: the MW of the analyte, the analyte polarity, the analyte concentration, and the complexity of the matrix (in the direct immersion sampling). Polydimethylsiloxane (PDMS) phase is for non-polar and volatile compounds; for low molecular weight, the carbon wide range/polydimethylsiloxane (CWR/PDMS) phase is preferred. The SPME fibers are used manually in combination with a holder that allows to handle safely the fiber, expose it, retract it when it is needed and injected in injector of the instrument where the desorbing of the analytes is obtained, typically a GC–MS. The holder itself it is not strictly necessary for the use of the fiber but it is essential to handle it safely.

Different fibers with different phases could be mounted on the same drone, to sample different volatiles; or different fibers of the same phase, that are exposed in different moments, to sample in different zones. Hangzhen Lan [3], in his doctoral dissertation "Development of Materials and Methodologies for Microextraction Techniques," makes an extensive and complete treaty that extensively shows various applications of SPME techniques.

1.2.1 SPME applications in forensics, CWA, TIC, pesticides, and contaminants.

SPME is a tool extensively used in forensic science, for sampling and analyzing volatile and semi-volatile compounds, by headspace or direct immersion. Some of the most notable applications of SPME in forensic science are the following. Furton et al. [4] have explored the detection and analysis of explosives and ignitable liquids from forensic specimen. In arson investigations, SPME can be used to extract and identify accelerants from fire debris. This technique helps in identifying the presence of ignitable liquids that may indicate deliberate arson [5]6. The ASTM has also published a standard practice for separation and concentration of ILRs from fire debris samples [7].

SPME can also be used to analyze food [8]. SPME is also widely used to analyze blood, urine, oral fluid, and hair in clinical and forensic toxicology [9]. SPME is also used in environmental analysis to study soil adsorption coefficients of organophosphorus pesticides [10].

SPME has also been used for the analysis of CWAs; Popiel et al. [11] focused on selecting appropriate SPME fibers and optimizing extraction conditions (time, temperature, and sample preparation). For this goal, several types of CWAs, including nerve agents (e.g., sarin and soman) and blister agents (e.g., sulfur mustard), were analyzed, as well as several SPME fibers, were evaluated for their

Table 3 Recommended fiber based on the characteristic of the analytes

Analyte type	Recommended fiber
Gases and low molecular weight compounds (MW 30–225)	75 µm/85 µm Carboxen/polydimethylsiloxane
Volatiles (MW 60–275)	100 µm polydimethylsiloxane
Volatiles, amines, and nitro-aromatic compounds (MW 50–300)	65 µm polydimethylsiloxane/divinylbenzene
Polar semi-volatiles (MW 80–300)	85 µm polyacrylate
Non-polar high molecular weight compounds (MW 125–600)	7 µm polydimethylsiloxane
Non-polar semi-volatiles (MW 80–500)	30 µm polydimethylsiloxane
Alcohols and polar compounds (MW 40–275)	60 µm Carbowax (PEG)
Flavor compounds: volatiles and semi-volatiles, C3-C20 (MW 40–275)	50/30 µm divinylbenzene/Carboxen on polydimethylsiloxane on a StableFlex fiber
Trace compound analysis (MW 40–275)	50/30 µm divinylbenzene/Carboxen on polydimethylsiloxane on a 2 cm StableFlex fiber
Amines and polar compounds (HPLC use only)	60 µm polydimethylsiloxane/divinylbenzene

extraction efficiency. The study optimized parameters such as extraction time and temperature to enhance the extraction efficiency of CWAs from different matrices, obtaining a high sensitivity and low detection limits for a wide range of CWAs.

Tucker et al. [12] compared different techniques for the identification of pesticides in viticulture and outlined the advantages and disadvantages of using the SPME, compared with other extraction techniques.

Hereinafter are resumed some of the most frequent applications of SPME and the recommended fiber (Table 3) [13]:

- Environmental analyses of water samples.
- Headspace analysis of trace impurities in polymers and solid samples.
- ppt odor analyses.
- Flavor analyses of food products.
- Forensic analyses of arson/explosives samples.
- Toxicology analyses: blood alcohol or drugs in urine/serum.
- Surfactants, other industrial applications.

In the following table, there is a list of the analyte type or class of compound and the suggested fiber.

The SPME must be screwed into a holder. The commercial holders are:

- For manual sampling use (manual holder)
- For automatic use in autosamplers (automated holder)
- For field sampling (SPME portable sampler) that allows to concentrate organics in the field and store them for transport to the laboratory.

The screw at the end of the fiber makes possible to mount a fiber on a customized holder.

Another important factor to consider is to use automated autosampler to analyze all the fibers that have been used during a campaign, to standardize and speed up the process. Dugheri et al. [14] in 2022 exhaustively considerate some commercial automated autosamplers for microextraction techniques.

In summary, SPME is a valuable tool for detecting low levels of contaminants in environmental samples. However, to achieve the best results, careful consideration must be given to the choice of fiber, extraction parameters, and analysis conditions. The use of an autosampler can make the analysis more reproducible, faster, and less time-consuming.

By optimizing and combining these factors, SPME can provide rapid, sensitive, and reliable analysis of CWAs, TICs, explosives, pesticides, and volatile contaminants in general.

1.3 Introduction to UAVs.

Unmanned Aerial Vehicles (UAVs) or Remotely Piloted Aircraft Systems (RPAS), commonly called drones, have emerged as versatile and valuable tools across a wide range of industries and applications. These aerial vehicles, typically operated remotely or autonomously, offer unique capabilities for data collection, surveillance, monitoring, and aerial imaging. Initially developed for military purposes, UAVs have rapidly expanded into civilian and commercial domains, revolutionizing sectors such as agriculture, construction, filmmaking, environmental conservation, and public safety. Equipped with advanced sensors, cameras, and communication systems, drones provide unparalleled access to aerial perspectives, enabling efficient and cost-effective solutions to various challenges. As UAV technology continues to evolve and becomes more accessible, cheaper; its potential for innovation and impact is unlimited.

In the next paragraph, the principles of European and Italian legislation on drones will be presented, along with an addendum of a decree regulating the use of drones by Italian police forces, to explain in which framework the drones can fly according to the European and Italian law.

1.3.1 Legislation on UAVs [15], 16

The European legislation applies to all European countries. The (EASA) European Union Regulations include:

Commission Implementing Regulation (EU) 2019/947 of May 24, 2019, on the rules and procedures for the operation of unmanned aircraft, which outlines the general rules for drone operations within the EU, including the classification of operations and operator requirements.

Commission Delegated Regulation (EU) 2019/945 of March 12, 2019, on unmanned aircraft systems and on third-country operators of unmanned aircraft systems, which sets the requirements for drone design, production, and market placement.

For operations falling under the provisions of Article 2, paragraph 3(a) of Regulation (EU) No. 1139/2018 on common rules in the field of civil aviation and establishing a European Union Aviation Safety Agency, and for aspects remaining within the competence of the Member State, in Italy, ENAC has published the UAS-IT Regulation on Remotely Piloted Aircraft Systems (RPAS) (in Italian "Regolamento Mezzi Aerei a Pilotaggio Remoto"), applicable from December 31, 2020. This regulation specifies the national requirements for the operation of drones in Italy, aligned with EASA regulations but with additional national provisions.

In Italy, there is also the D-Flight Portal, which is the official platform for drone registration, flight authorizations, and airspace information. Compliance with ENAC regulations is essential.

Compliance with the European Union (EASA) Regulations and national regulations is crucial for drone operators to avoid fines and legal consequences while maximizing the benefits of drone technology in various sectors, including photography, videography, agriculture, infrastructure inspection, and public safety. The regulations cover several aspects of drone use, including classification, registration, pilot training and certification, operational and geographical restrictions, safety measures, insurance requirements, and privacy and data protection.

The use of drones by Police Forces in Italy is regulated by the Decree of the Ministry of the Interior on the “Methods of use of remotely piloted aircraft by the police forces,” [17], addressed to the police forces referred to in Article 16 of the law of April 1, 1981, No. 121 [18]. This decree, in agreement with the Minister of Defense and the Minister of Infrastructure and Transport, after consulting the National Civil Aviation Authority, regulates the methods of use of remotely piloted aircraft by the police forces for territorial control for public security purposes, particularly for combating terrorism and preventing organized and environmental crime.

The extensive use of drones for urban and public security purposes must also consider precautions for the rights and fundamental freedoms of the individuals concerned in light of Regulation (EU) 2016/679 and Legislative Decree No. 51/2018 on police data processing.

This means that each police force must conduct a privacy impact assessment, including a comprehensive risk analysis to measure and manage potential negative impacts on the rights, fundamental freedoms, and dignity of the individuals concerned. This will require particular commitment given the peculiarities (primarily aerial footage) of the tool (the drone) used to capture images that could potentially record private domiciles.

Nevertheless, European and Italian legislation on drones allows their use quite freely in the open category, provided there are no people present. This is typical in scenarios such as forensic investigations, the release of TICs, or suspected use of CWAs. Detection of pesticides is usually conducted in agricultural fields. The potentially dangerous scenarios in which we are studying the possible deployment of drones typically do not involve people because the area has been evacuated or is otherwise uninhabited due to danger. Therefore, drone use in these situations complies with safety and legal requirements.

The recent agreement signed between the Italian police forces and ENAC (Italian Civil Aviation Authority) (on May 15th) regulates the use of drones in various operational scenarios, in order to provide even more effective responses in the fight against crime.

1.3.2 Integrated uses of sensors and sampling systems with UAVs

Integration with drones and MOX and PID: Review of the literature In an extensive article, Di Giovanni et al. [19] present practical applications related to the detection of different agents, in which a drone with sensors can be used. Basically, two main field of interest are envisaged by the authors: civil and military, as the drones could be used in conventional and non-conventional risk scenarios. Among some other kind of chemical sensors/analyzers, thanks to their sensitivity, fast response, lightweight, and low cost, PIDs and MOXs are the most used sensors in CWA applications and for first responders. The integration of chemical sensors on drones gives them the capability of being employed in the need of detecting VOCs, CWAs, environmental monitoring, TIC, CBRNe incidents, illegal spillage of hazardous substances, or the attempt of destruction by fire of unauthorized waste. In the article, they also talk about integrating scintillators, ion mobility spectrometry, and gamma radiation identifier on drones, but these categories of equipment are more expensive than MOX, PID, and SPME and are far beyond the scope of this dissertation.

In this article, Martellucci et al. [20] study the practical advantages of using a drone swarm equipped with chemical sensors, such as PID and/or MOX, for the accurate monitoring and early detection, localization and identification of a chemical release. Some examples of finding a plume of potentially dangerous chemical with a single drone and with a drone array are presented, showing how the drone swarm is more effective, in terms of localizing and in terms of time, that in the case of toxic substance release is crucial for the safety of the environment and of the living creatures. In the so-called Gas Source Localization task, that consists in finding the gas emission source point, a drone swarm could be more effective than a single drone. Moreover, not only the localization but also the plume tracking plays an important role in the case of toxic substances release. The authors then theorized a scalable approach to these tasks, using different kinds of drone equipped with different sensors, to perform at the best; for example, very fast drones to rapidly follow the plume, while heavier and more equipped drones could identify the substances.

Integration with drones and SPME: Review of the literature While the literature of use of chemical sensors or chemical traps on drones is very rich, the literature of use of SPME on drones is very limited. This paragraph permits to review briefly all the scientific articles where a SPME is used in combination with a drone; it must be noted that no article has been found that deals with the combined use of PID and/or MOX sensors and SPME.

In the 2019 study by Ruiz-Jimenez et al. [21], use aerial drones equipped with miniaturized air sampling systems, (SPME Arrow and ITEX), for environmental monitoring. It is the first occurrence of the use of SPME carried on a drone, in a scientific article. The authors developed a drone equipped with SPME and ITEX sampling systems. ITEX is the name of a sampling technique that is essentially a hollow tube filled with a sorbent, with higher sorbent capacity of a SPME. The ITEX tube has an extremity with a needle with a hole where the air is conveyed by a pump attached to the other extremity. The authors executed several samplings with both systems, in three different places: a soccer field area, and two places difficult to reach with other means: a forest and a wetland area in Finland. They performed the experiments with a two kilos drone. The sampling time was 30 min for experiments with only one collection per flight, and 15 min for experiments with two sampling per flight. The time was chosen because of the 35 min maximum per flight. They equipped the drone with a system to hold the SPME Arrow and capable to open and close it by remote, when it is needed.

The study, other than highlighting the advantages of using drones equipped with SPME (up to four of them mounted simultaneously), demonstrates that the use of SPME on a drone is feasible also in zones difficult to reach, and can be effective in capturing VOCs of different chemical species; in fact, they were able to identify several chemical compounds of the following classes of compounds: aldehydes, ketones, alcohols, hydrocarbons, acids and derivatives, nitrogen containing compounds, and sulfur containing compounds.

In 2019, study by Cheng et al. [22] developed and tested a sampling system with a microextraction needle trap device to collect organic vapors. They used a telescoping needle trap device, capable of being out of the air stream produced by the drone propellers. They used an active sampling device: a pump would assure a flow of air passing through the needle. For the purpose, they used a flow simulator software to predict the zone with less or no turbulence intensity and performed some tests with dry-ice spray to visualize the air stream. They were able to detect toluene, ethylbenzene, and p-xylene.

In the 2020, study by Grandy et al. [23] presented the application of a sampling system of the water, carried out with a SPME mounted on a drone. Not being the principal goal of this work to talk about water sampling, this article was reported to show the state of the art of drone-SPME systems and to show their flexibility since that they can use the SPME to work also by immersion.

Lan et al. [24] in 2021 tried a quantitative and semi-quantitative approach for VOCs in the atmosphere. They performed several tests, with SPME (passive sampling) and ITEX (active sampling). The drone was equipped with SPME arrow and ITEX.

The research developed a method for the quantitative analysis of VOCs, and the sampling was performed at different heights, to outline the difference of the sampling position. The time of sampling and the influence of the turbulence of the propellers were investigated, finding that the best position for the passive sampling performed by the SPME was under the drone where it was less influenced by the turbulence. Moreover, they found that SPME could give a recovery response comparable with the ITEX, that is, the gold standard for VOCs or air pollutants quantitation.

In 2022, the University of Caldas [25] launched a project to investigate the use of drone-SPME technology for controlling and mitigating atmospheric emissions from both stationary and mobile sources in the metropolitan area of Medellín, with the aim of enhancing air quality management in urban environments.

In 2022, Chen et al. [26] gave rise to research where an array of drones is deployed to sample air pollutants with a SPME. They performed several tests for different chemicals and were able to quantitate the pollutants and map their spatial distribution. They tested the signal responses of VOCs obtained with different sampling methods, under different conditions and exposing time, under different velocities of airflow, under different sizes of drones, varying the sampling time, and the quantitation of chemicals extracted on the SPME fiber.

Their choice was to mount the fiber on the side of the drone, to seal the SPME with a septum, while non-sampling, to preserve the collected chemicals from loss or contamination, and to have a great air flow on the SPME.

The technology of mounting one or more SPME on a one or more drone is for sure a powerful tool for environmental monitoring, giving comprehensive and immediate responses to air quality issues. Nevertheless, the extensive research on the literature has not showed any occurrence of articles describing the coupling of chemical sensors and SPME mounted on a drone.

2 Materials and methods

The experiments were carried out to explore the possibilities of mounting a SPME fiber on a drone and pursued three main purposes.

a) To demonstrate that even a small drone can fly with a complete SPME fiber and holder that demonstrates that bigger drones with more payloads can easily transport an array of SPME.
b) Developing a cheap and reproducible experimental setup that could be used for testing, in the same controlled conditions of temperature and chemicals concentration, the SPME fiber, and the sensors.
c) Test the experimental setup and prove that is reliable and can be easily used, making an experiment using a SPME to adsorb some test molecules and detect them with a GC–MS.

All the experiments were performed in the forensic laboratory of the Interregional Cabinet of forensic Police for Piedmont and Aosta Valley of Turin.

To perform the experiments, the following material was used:

a) micro-drone to carry the SPME holder.
b) GC–MS to perform the analysis.
c) SPME holder and a fiber to perform the extraction.
d) Chemical test mixture.
e) Syringe to take the right amount of chemical mixture.
f) A nominal 1,5 L jar with screwcap and an aluminum foil, where to put the chemical mixture and performing the extraction with the SPME.
g) Oven, to keep the jar at a controlled temperature and perform the chemical extraction at controlled temperature.

2.1 Drone

The drone used for the test is a micro-drone, brand Drone Explorers of the weight of 77 g. This drone was chosen because is a basic and low cost drone, it is lightweight, it can be flown without restrictions and does not need a professional pilot.

2.2 GC–MS and chromatographic conditions

The instrument used for the analysis of the SPME is a GC–MS (8890 GC System coupled with a 5977B MSD Agilent Technologies), equipped with a HP5MS column and used helium as carrier gas. The chromatographic and MS conditions are listed in Tables 4 and 5.

The chromatographic conditions are derived from a method used to detect hydrocarbons, after the absorption on a SPME from fire debris, and it has been tested to be suitable for the purpose. The proposed chromatographic conditions have been already tested with complex hydrocarbon mixes and allow the detection of a wide range of hydrocarbons derived from a petroleum derived mixture (like gasoline for example), that allow a separation of the compounds, good enough to allow a classification of the nature of the petroleum derivative into the ignitable liquid classification scheme of the ASTM 1618 (Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography–Mass Spectrometry). The source and quad temperatures chosen are the standard ones for many GC–MS applications, as the 70 eV electron energy. The full scan from mass 30 to mass 250 allows to detect a wide range of compounds in the hydrocarbon class.

2.3 SPME holder and fiber

The SPME holder used for the experiments is a manual holder (Catalog number 57330-U, Supelco, Bellafonte, PA) in which the fiber must be mounted and must be conditioned in the GC–MS, before being used.

Other holders exist, that can be used with the automated system, or manual for field sampling.

The SPME fiber used for the experiment is a green code color fiber, PDMS, 7 μm, for manual extraction, part number 391896304, lot 57,272, Agilent Technologies. This is a fiber suitable for non-polar high molecular weight compounds (MW 125–600).

2.4 The chemical mixture

The chemical mixture is a FID-TCD Performance Evaluation Sample Kit, in a closed ampoule, Agilent part number 5080–8842, lot 0006689316, consisting of a mixture of three linear hydrocarbons, n-tetradecane ($C_{14}H_{30}$), n-pentadecane ($C_{15}H_{32}$), and n-hexadecane ($C_{16}H_{34}$) in hexane, in the following purities and concentrations.

a) *n*-tetradecane 0.218 g/L ($\pm$ 0.5%), purity 99%
b) *n*-pentadecane 0.218 g/L ($\pm$ 0.5%), purity 99%
c) *n*-hexadecane 0.218 g/L ($\pm$ 0.5%), purity 99%

Table 4 Instrument control parameters

Chromatographic conditions	
Control information	
Sample inlet	GC
Injection source	Manual
Mass spectrometer	Enabled
GC	
Run time	28 min
Post-run time	0 min
Oven temperature	
Set point	On
Initial	60 °C
Hold time	1 min
Post-run	0 °C
Program	
#1 Rate	20 °C/min
#1 Value	260 °C
#1 Hold time	10 min
#2 Rate	10 °C/min
#2 Value	300 °C
#2 Hold time	3 min
Front SS inlet	He
Mode	Split
Heater	On 250 °C
Pressure	On 8.2317 psi
Total flow	On 14 mL/min
Septum purge flow	On 3 mL/min
Pre-run flow test	Off
Gas saver	On 20 mL/min after 2 min
Split ratio	10:1
Split flow	10 mL/min
Liner	Agilent 19,251–60,540: Inlet liner, split, straight, with glass wool

Table 5 Single quadrupole acquisition method

MS parameter report	
Tune file	etune.u
Ion source	EI
Source temperature (°C)	230
Quad temperature (°C)	150
Fixed electron energy (eV)	70.0
Acquisition type	Scan
Solvent delay (min)	0.20
Gain factor	1
Scan time segments	
Time	0.20
Start mass	30
End mass	250
Threshold	150
Scan speed	1562 [n = 2]

d) hexane, purity 99,6%

The mixture was chosen for the following reasons:

a) It is a mixture commonly used to check the performances of Flame Ionization Detectors (FID), and it can be easily purchased by every laboratory for further tests.
b) The mixture contains a mix of hydrocarbons that is the class of compound that is researched as Ignitable Liquid Residues (ILr) consequently to an arson, in forensic applications.
c) The compounds present in the mixture are suitable for being detected by the SPME used in this work.
d) In further tests with PID and MOX, this mixture could be used to compare the response of the chemical sensors and the SPME.

2.5 The syringe

The syringe used for sampling the chemical mixture is a 10 µl Agilent Gold Standard Syringe, Agilent Technologies PART #5181–1267, with a subdivision of 0.2 µl. This is a standard syringe used in the GC high precision autosampler or for manual injection in GC. The producer guarantees for this syringe and accuracy of ± 1% and a precision of ± 1%.

2.6 The container

The container user for the experiment is a 1,5 L nominal glass jar used for food preserving and canning, complete with a screwcap that can close it tight, with its metallic screwcap. The screwcap is internally covered with a layer of rubber that ensures the tightness. Nevertheless, the rubber can absorb the components of the chemical mixture so, a layer of aluminum foil is inserted between the jar and the screwcap. This ensures the tightness during the thermostatic phase of the experiment and avoids the chemicals to be in contact with the rubber of the screwcap. In this way, the jar and the screwcap can be used as many times as is needed, providing that the glass jar is washed, and the aluminum foil is substituted.

To perform the experiment, the screwcap was punctured to make a small hole in the center that is used to insert the SPME holder at the moment of the sampling.

The effective volume was calculated weighting the jar at 20 °C, empty and full of demineralized water, weighted with the screwcap and with the aluminum foil. The volume, used in the calculations for the concentration of the analytes, resulted to be 1668 cm^3.

To equip a drone with a SPME, one standard holder can be used if there are no limits on the payload of the drone. The classic holder plus the fiber, accounts for 35.4 g, while the fiber only weights 1.1 g. Nevertheless, the screw at the bottom of the SPME fiber allows to design and produce custom-made holders, for example, with a 3D-printer.

In the simplest configuration, a drone could be equipped with an already exposed fiber and a single chemical sensor. After the flight, analyzing the fiber would reveal all the substances captured during the flight. This type of drone can be used until it reaches an area of interest, indicated by a positive signal from the sensor. At that point, the sampling is considered complete, and the drone can be retrieved. In this case, the analysis of the fiber provides a record of all the chemicals encountered during the flight, but it would not be possible to determine the exact location where a specific chemical was absorbed. This configuration is limited but still informative if the flight is restricted to a specific area.

If we want instead the fiber to be exposed only in a certain zone and not before and not after, the fiber must be equipped by a motorized opening for exposing and retracting it when desired. For example, the fiber could be exposed only when the PIDs or the MOXs mounted on the drone give a signal.

Conversely, a drone can be equipped with multiple chemical sensors and several SPME fibers that remain enclosed during the flight. A small device can expose the fibers only when one or more chemical sensors detect a positive signal and then close them once sampling is complete. This method allows for controlled sampling only when needed. The analysis of the fibers can be linked to specific sampling areas, enabling multiple different samplings with various fibers in different zones.

This integrated system with SPME fibers, unlike some of those already in use, is lightweight (unlike canisters that have been mounted on drones) and does not require pumps, such as Tenax or charcoal tubes that have also been used on drones. Another consideration is that before mounting an SPME fiber on a drone, the turbulence generated by the rotor blades must be studied to prevent the rupture of the SPME and ensure correct sampling in steady conditions.

3 Experimental setup and results

3.1 SPME integrated with a drone: experimental setup and results

The first test was performed using a lightweight drone. The scope of the test is to demonstrate that the SMPE with his holder is lightweight and can be mounted on a drone, to be used for sampling purposes. The SPME mounted on the holder accounts only of 35.4 g, while a single fiber accounts only of 1.1 g. Moreover, since at the end of the fiber there is a screw, this can be attached to a different holder that has to be produced specifically, that can be also produced to hold more fibers.

Fig. 1 Screenshot of the drone flying with the SPME holder on the bottom

The fiber with its standard holder was attached at the bottom of the drone, in the center. The drone could easily accommodate the fiber in that position without touching the ground while the UAV landing since the SPME holder is thinner than the four feet of the drone.

The drone could easily accommodate the fiber that did not affect its stability neither the flight of the drone that was carried out without evident problems.

Also the maneuverability of the drone was not affected, since the drone could perform aerial movements (right-left, front-back, and up-down) without being heavily affected by the SPME, demonstrating the feasibility of mounting a SPME fiber even on a small drone.

The movements of the drone did not break the fiber after the two sample flights.

In this configuration, it was not performed a test of sampling with the SPME to not to risk breaking the SPME since not having a device to extract the SPME and retract it, should have been completely extracted during all the flight; secondly, because there was not a target compound in a controlled environment to be sampled.

A video of the two flights was performed. In Fig. 1, a screenshot of the video shows the drone flying with the SPME holder on the bottom.

3.2 SPME tests in static conditions: experimental setup and results

The second test was conducted to evaluate the suitability of the setup to perform experiments in controlled conditions of temperature and humidity. The test was performed in the most basic way, only with the temperature control, but could be expanded using other tools that could be easily inserted through the screwcap of the jar that is easily modifiable (Fig. 2).

The screwcap of the jar could be then modified to accommodate also the chemical sensors such as MOX or PID, so after inserting the chemical, it could be sampled with the SPME and the chemical sensors response could be recorded. Also, a thermohygrometer could be easily fitted, to measure the temperature and the humidity, considering that are parameters that affect the chemical sensors.

To perform this experiment, 1 µl of the sample mixture FID-TCD Performance Evaluation Sample Kit was taken with the 10 µl syringe and inserted in the opened jar that was closed immediately with an aluminum foil and the screwcap, closed tightly. With this dilution, each component of the mixture results of a concentration of 0,13 ppb.

Then, the jar was put in an oven at 30 °C for 15 min. The temperature was chosen to simulate the air temperature and higher respect the room temperature to be sure that the oven could work properly; after 15 min, the SPME was exposed into the jar, through the hole, piercing the aluminum foil.

The results of the sample analysis gave three peaks (Figs. 3, 4, 5, 6) corresponding to the standard mixture, as they were correctly identified using the library Nist20.L. What is notable in this analysis is that the concentration of the three hydrocarbons is of 0.13 pbb, and their peaks are really clear, well above the baseline. The three peaks are slightly different (the peak corresponding to the molecule with lower MW is smaller). To understand if this is due to the different sensibility of the instrument used for the analysis performed by the producer, other experiments must be performed. The smaller MW compound is also more volatile than the others with higher MW, so an effect of higher rate of evaporation (and passing through the hole that is not gas tight or evaporation when deposited in the jar) could be inferred.

The test could be modified putting a septum on the hole, so the jar becomes gastight and during the sampling of the jar there is no space left to let the volatiles to go away.

Nevertheless, the experiment proved that this an effective method to test in steady and reproducible conditions the response of the SPME to different compounds, in a closed container where the exact concentration can be calculated; this method could also easily fit some chemical sensors into the screwcap, so the tests could be performed simultaneously on SPME and PID and/or MOX

Fig. 2 The jar with the 1 µl of the test mixture, closed, in the oven, during the 5 min sampling at 30 °C (right). After the 5 min of sampling, the SPME was injected into the GC–MS to perform the analysis, where the desorbing time was of 1 min

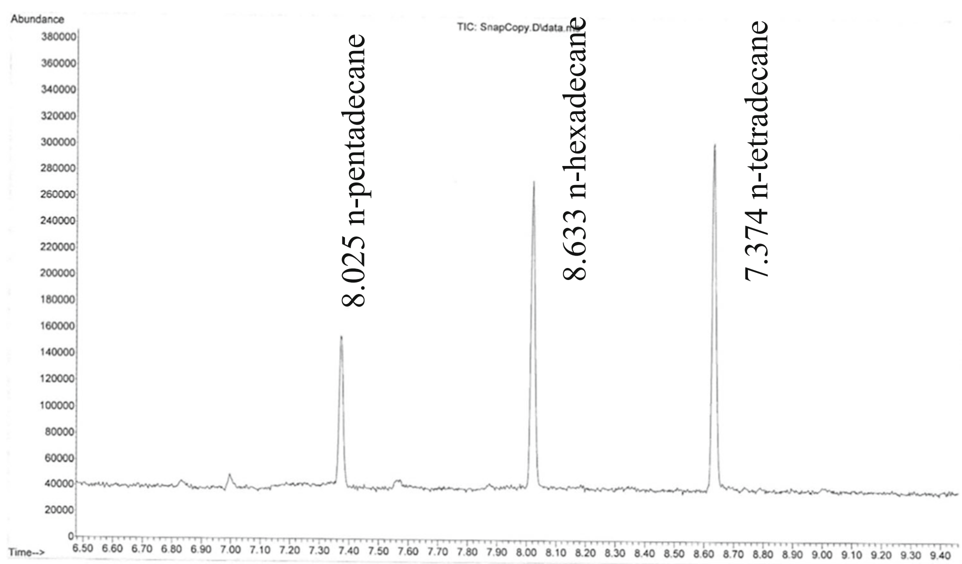

Fig. 3 The total ion count chromatogram of the chromatographic analysis of the SPME desorbed after the sampling. Each hydrocarbon has a concentration of 0,13 ppb

sensors, comparing the different responses of the different devices. Also, a thermohygrometer could be easily fitted in the screwcap, so the humidity (that is known to be a influencing factor for chemical sensors) and the temperature could be measured.

4 Conclusions

While the use of chemical sensors on drones is widely used and trusted, and it is common to encounter sensors mounted on drones even in scientific publications, the use of SPME is currently limited to laboratory or field use, but not much with drones. Only a few scientific articles have been published using SPME mounted on drones and no one in combination with chemical sensors. This could be due to different main factors: first, because SPME requires an automatic trigger to be opened; second, because of the fragility of the fiber; and third, because the classical SPME could not be sealed after conditioning and subsequently, after sampling. Mounting one classical SPME fiber on a drone, even with motorized exposure only when needed, could not completely prevent contamination due to the shape and the way the fiber was retracted into the needle, which has a slight opening. The second and third factors are

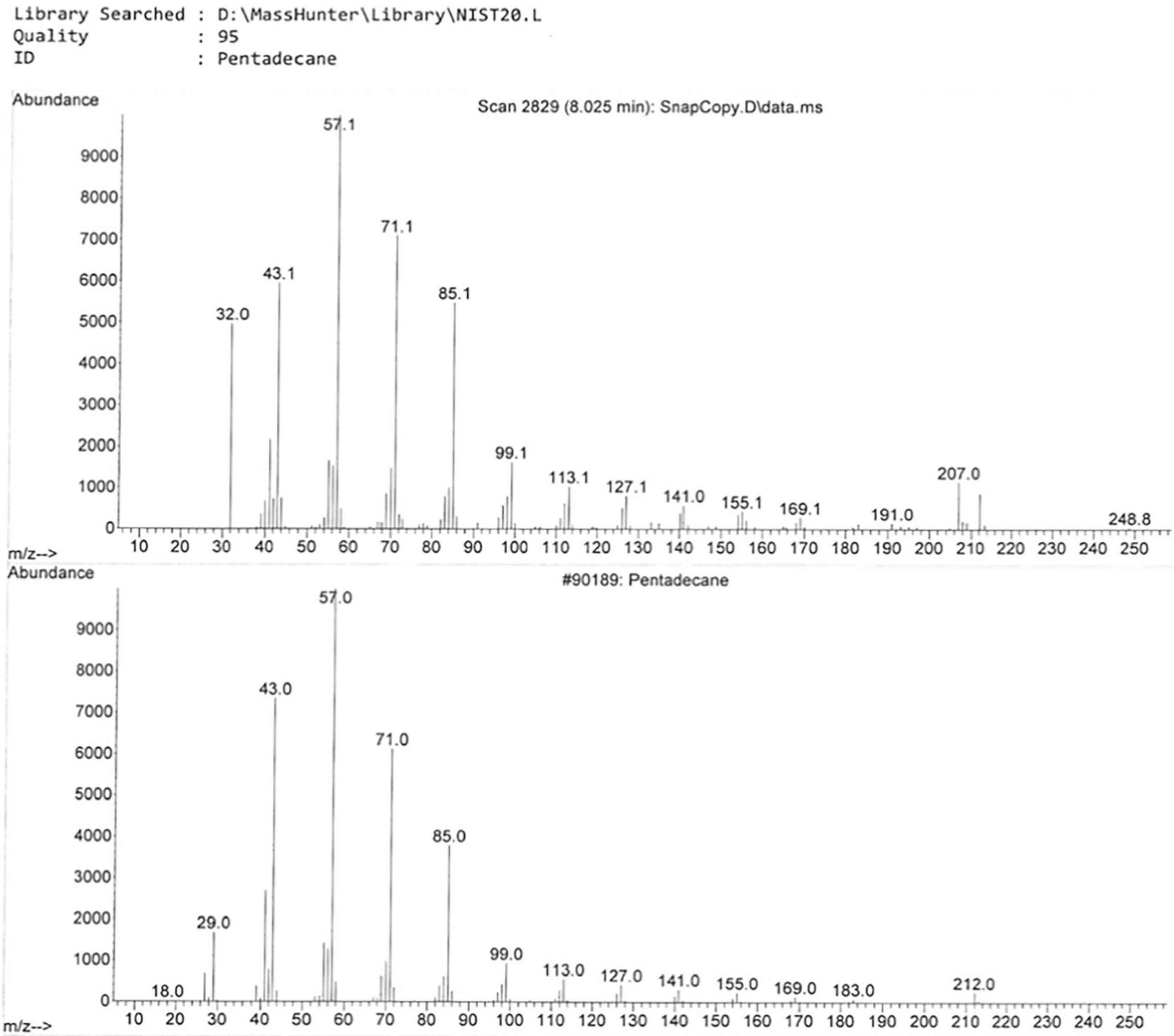

Fig. 4 The peak at 8.025 correctly identified with a Nist20 library as pentadecane

now being overcame by the new SPME arrow, which is rugged and is completely closed after retraction, even if in this work we proved that also the classic SPME could be mounted and transported on a drone. Another reason, in this case ontological one, could be that use of SPME is a specific target for chemists, while drones and/or chemical sensors are generally studied by engineers, so the two worlds are usually not considered together; in fact, the idea for this work emerged from the multidisciplinary approach of the master's program on CBRNe event protection of the University of Rome Tor Vergata.

This study has exhaustively shown the idea on how the civil use of UAVs, coupled with non-expensive and well-established electrochemical sensors such as PID and MOX, along with the lightweight SPME sampling technique, can be a useful tool for performing online screening coupled with air sampling. The coupling of online screening offers the advantage of responsiveness to various volatile compounds, such as pesticides, warfare agents, pollutants, combustion residues, and dangerous or poisonous chemicals released during industrial disasters or in situations requiring unmanned sampling for safety or investigation purposes.

However, while chemical sensors can provide an immediate alert response, they lack specificity and have some drawbacks: The chemicals cannot be uniquely identified, and there are possibilities of false positive responses. In contrast, the sampling technique acts as a trap for the chemicals, and subsequent laboratory analysis via GC–MS can reveal the exact nature of the encountered compounds. SPME sampling could be automated and triggered when the electrochemical sensors give a positive response, allowing sampling only when necessary, and in a specific zone that can be determined. The lightweight nature of SPME is advantageous, as UAVs can be equipped with multiple SPME devices with different sorbent capacities for different classes of compounds or multiple SPME devices of the same type for multiple sampling in different moments or places.

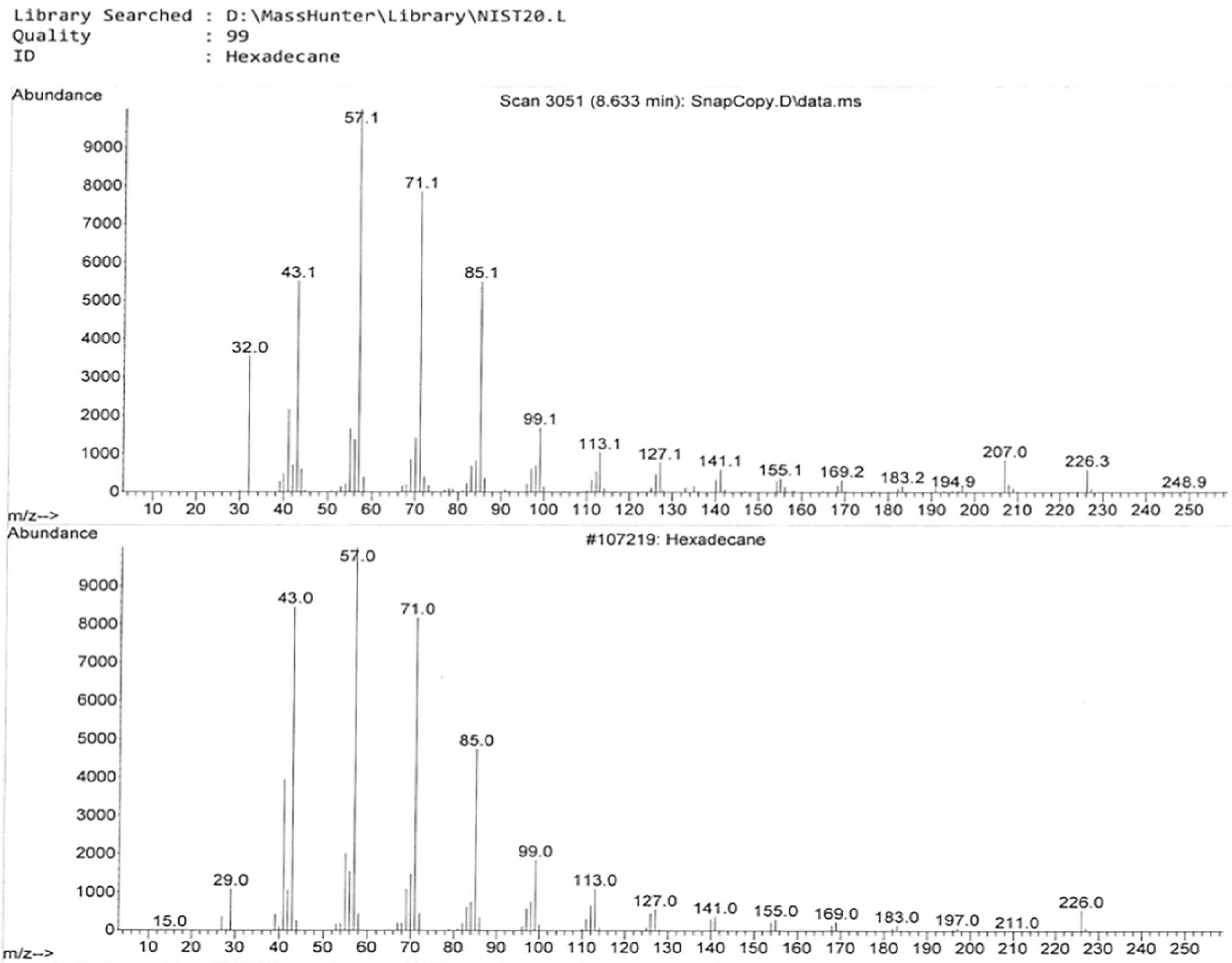

Fig. 5 The peak at 8.633 correctly identified with a Nist20 library as hexadecane

SPME fibers need to be activated before use, which is a relatively simple task. Then, it must be mounted on the UAV and then unmounted after use for analysis on a GC (coupled with different detectors). There are also commercial autosamplers for SPME fibers that allow automatic injection into the GC for subsequent analysis. The UAV must be equipped with a small device to open and close the fiber compartment at the right moment, which can be performed remotely by the UAV operator or automatically after the electrochemical sensors detect notable substances.

This integrated approach can be used when the compounds are volatile, and they can be absorbed and retained by the SPME fibers; several fibers have been produced so far, and a lot of compounds can be retained and analyzed. For nonvolatile compounds, other approaches should be used, like sampling the soil or the water, that could then be analyzed as well with SPME.

The principal drawback of this system is that the analysis must be conducted in a laboratory and is not immediate unless a portable (but expensive) GC–MS is used. Light gases are generally not retained by the fibers, but for poisonous gases, other types of sensors can be used, and gas release scenarios may differ from scenarios involving chemical warfare agents (CWA), toxic industrial chemicals (TIC), and volatile organic compounds (VOC), that can be revealed by the drone-sensor-SPME. In the near future, it is possible that miniaturized chemical analyzers could be fitted on UAVs, but today this approach is expensive and not fully scanned. Nonetheless, the use of lightweight and inexpensive SPME, followed by a GC–MS analysis, remains a promising and low-cost approach, that in the case of the loss of the drone itself does not represent a significant loss. This integrated approach is simple, lightweight, and inexpensive and could be deployed and used worldwide, with the possibility of deploying the UAV in dangerous or not easily accessible zones. The UAV can also be fitted with a camera and a GPS to locate exactly where the sampling has been made, crucial aspect in case of forensic activity.

No scientific articles that deal with the combined use of PID and/or MOX sensors and SPME on a drone have been found, confirming that this is an innovative approach deserving further exploration through combined testing.

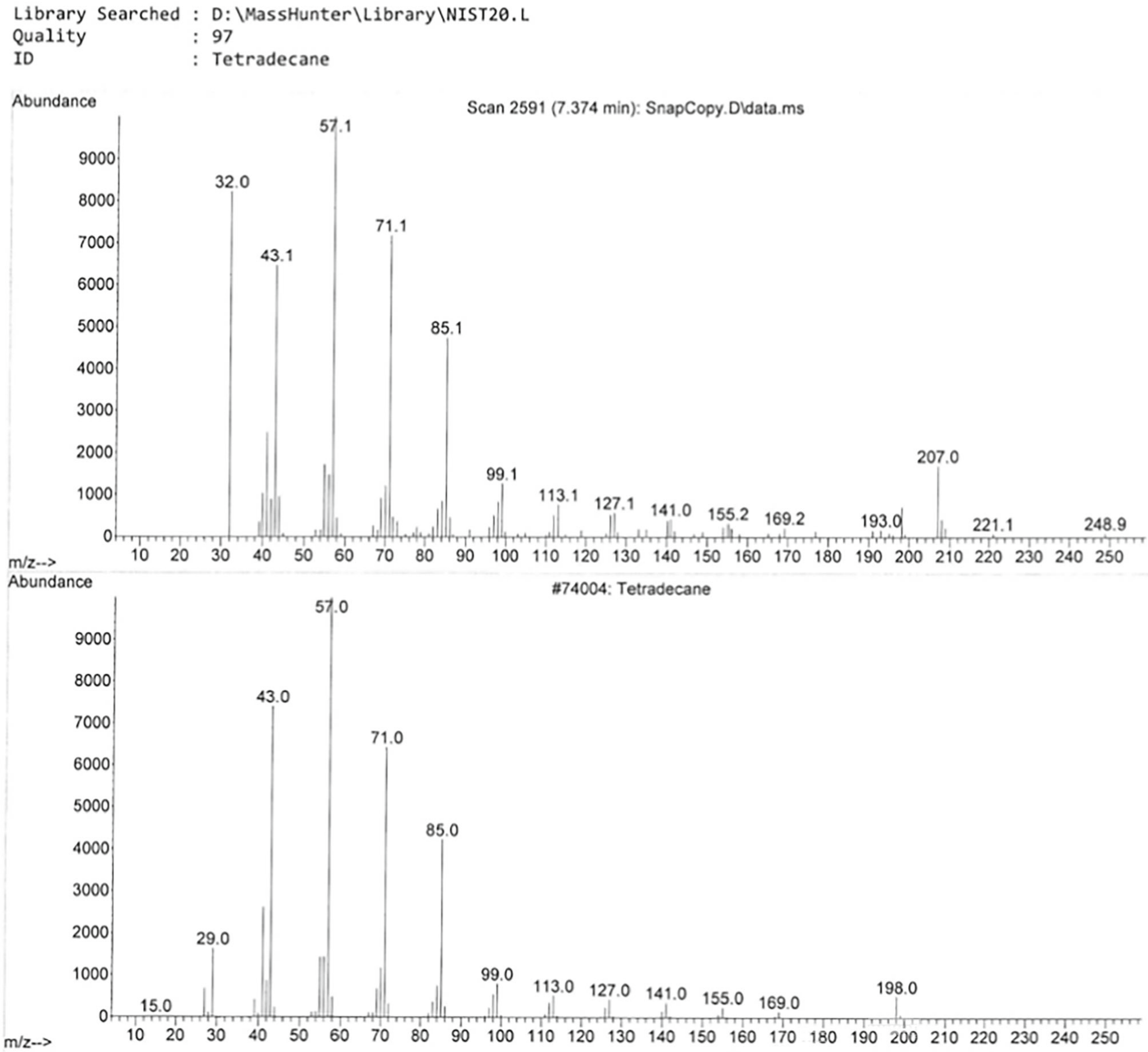

Fig. 6 The peak at 7.374 correctly identified with a Nist20 library as tetradecane

As being an innovative approach, some tests were performed to prove the feasibility of mounting a SPME even on a small drone, and on an equipment that was theorized and then proved to be effective to be as a possible device to be used as a bench test for SPME and chemical sensors.

Two kinds of test were performed:

a) With a very light micro-drone that was flown twice to show that the SPME fiber can be easily transported by it. If the SPME is mounted without affecting the barycenter of the drone, it does not affect its stability and maneuverability. In the simplest approach in which there is no need to extract and retract the fiber, because is mounted completely exposed, even several fibers could be mounted on a single small drone.
b) The second experimental part was performed to prove that a cheap container, modified on purpose to fit the SPME, could be used to test the fibers and other sensors, as they could be exposed to a known concentration of chemicals, in a steady and reproducible condition.

Future applications

Future perspectives should address to perform quantitative analysis on chemical substances and pollutants coupling chemical sensors with SPME and the versatility of the drones. This configuration would allow to have a direct detection through the chemical sensors; then, when a signal from a sensor is detected, the SPME could be exposed to sample the air. At the return of the drone/drones, the analysis on a GC–MS would show the chemicals and their amount. As it could be easily argued, forensic, environmental, industrial, and safety applications of such kind of system are infinite, both in civil and in military fields. To reach the best performance of these systems, it would be necessary to perform quite a big quantity of tests, considering that in a such complex system, a lot of parameters must be controlled and optimized. This work showed a simple and cheap system that it could be used to test both SPME and chemical sensors in controlled conditions, and this could spare hours of tests with the drones. The main parameter that should be investigated with a system as the one described in this work, for both SPME and chemical sensors, are:

a) Response at different concentrations;
b) Response at different temperatures;
c) Response at different humidity;
d) Response at different exposure times.
e) Response of different fibers/sensors on different chemicals.

After a systematic approach on these parameters, other parameters, with the use of the drone, must be considered:

a) The propeller flow stream;
b) The effect of the stream on the position of the SPME and of chemical sensors.
c) The sampling with a drone array, to map a zone of interest.

That said, some more work must be accomplished with drones combined with chemical sensors and SPME, both for testing and making it operational.

We strongly believe that the proposed use of equipping UAVs with chemical sensors and SPME would be a contribution to improve safety in CBRNe scenario, in dangerous situations or when handling hazardous chemicals, enhance the detection of substances of forensic interest, and thereby support the community and serve justice.

Bearing in mind that there are no single sampling methods that can detect all the compounds, the combination of PID and MOX sensors with SPME fibers on UAVs offers significant advantages in terms of real-time detection and accurate laboratory analysis. This innovative integrated approach herein proposed, would address the limitations of each individual detection component, providing a robust solution for the detection and identification of volatile compounds, also addressing the sampling in safe conditions or in non-otherwise accessible areas.

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Authors contribution All authors contributed to the study conceptualization and design. Material preparation, data collection, and analysis were performed by Morela Strano. Pasquale Gaudio, Daniele Di Giovanni, Luca Martellucci, Mariacristina Pigro, and Damiano Ricci supervised the work. Morela Strano wrote the first draft of the manuscript. All authors commented, edited, and reviewed subsequent versions of the manuscript. All authors read and approved the final manuscript.

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Data Availability Statement The methodology, the materials, and the conditions of the experiments developed in this work are entirely mentioned in the text of the article. No additional data are available for this work.

Declarations

Conflict of interest The authors have no financial or proprietary interests in any material discussed in this article. The authors have no competing interests to declare that are relevant to the content of this article.

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