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Dual Profiling of VOCs in Fire Scene Evidence Using GC-MS and LC-MS

Min Seok Suh[1,†], Jae Yoon Go[1,†], Min Uk Kim[1], Jeong-Hun Mok[1], Hee-Joung Lim[2], Eugeine Jung[2], Jong-Moon Park[3,*], and Hookeun Lee[1,*]

[1]College of Pharmacy, Gachon University, 191, Hambangmoe-ro, Yeonsu-gu, Incheon 21936, Republic of Korea
[2]Forensic Science Research Center, Police Science Institute, Korean National Police University, Republic of Korea
[3]Basil Biotech, 32, Songdogwahak-ro, Yeonsu-gu, Incheon, Republic of Korea

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Abstract : Accurate identification of volatile organic compounds (VOCs) generated at fire scenes is essential not only for determining the cause of ignition but also for environmental and toxicity risk assessments. In this study, we applied gas chromatography-mass spectrometry (GC-MS) and liquid chromatography-mass spectrometry (LC-MS) in parallel and introduced pretreatment methods suitable for each analyzer to expand the detection range of VOCs. Samples collected in adsorption tubes using thermal desorption were analyzed by GC-MS, and residual samples from the same adsorption tubes were extracted with solvents and analyzed by LC-MS. As a result, a maximum of 238 compounds were detected by GC-MS analysis, and 12 types of hazardous chemicals were commonly identified in all samples. In the LC-MS analysis, a total of more than 950 organic compounds were detected, of which 482 compounds were commonly identified in all samples. In particular, compounds with biological accumulation and endocrine disruption potential, such as 4–octylphenol and DEHP, were detected only in the LC-MS analysis. It was confirmed that the five compounds detected in both analyses could be used as cross-validation indicators. The sequential analysis strategy using GC-MS and LC-MS with the same collection tube improved both analytical reproducibility and efficiency, demonstrating its potential for application in various fields such as chemical accident response, forensic analysis, and environmental risk assessment.

Keywords : Volatile organic compounds (VOCs), GC-MS, LC-MS, fire scene investigation, forensic chemistry

Introduction

Modern forensic science uses a variety of analytical techniques to investigate criminal cases in a scientific and objective manner. In fire-related investigations, it is crucial to determine whether arson occurred, identify the type of ignitable materials involved, and reconstruct the sequence of events.[1] Among the key chemical indicators, volatile organic compounds (VOCs) released at fire scenes serve as essential clues for tracing the cause of ignition, and thus require precise analytical methods.[2] However, gaseous evidence containing VOCs is highly unstable due to extreme conditions at fire scenes such as heat, pressure, and airflow which can easily alter or degrade its chemical composition. Therefore, appropriate sample pretreatment and selection of analytical instruments are critical for ensuring accuracy and reproducibility in the analysis. In fact, gas chromatography-mass spectrometry (GC-MS) is widely used as a standard tool for qualitative and quantitative analysis of gas samples from fire scenes, and is particularly effective in detecting highly volatile and nonpolar compounds, including flammable liquid residues (ILRs). [3,4]

However, GC-MS has limitations in detecting high-molecular-weight, polar, or low-volatility compounds, and often encounters difficulties in distinguishing between target compounds and combustion-derived matrix interferences due to structural similarities. To overcome these limitations, liquid chromatography-mass spectrometry (LC-MS) has been employed as a complementary analytical technique. Volatile organic compounds (VOCs) are frequently characterized by their relatively non-polar nature, which hampers their effective ionization via the electrospray ionization (ESI) interface utilized in LC-MS. As a result, the detection sensitivity for VOCs with low molecular weight or weak polarity may be compromised. Nonetheless, prior research has indicated that VOCs are not solely composed of non-polar substances but also encom-

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†Both contributed equally.
*Reprint requests to Hookeun Lee
https://orcid.org/0000-0002-0696-8421
E-mail: hklee@gachon.ac.kr

pass compounds exhibiting significant polarity.[5,6] Accordingly, the present study applied LC-MS coupled with ESI to enhance the analysis of polar and semi-volatile combustion products that are inadequately detected by gas chromatography-mass spectrometry (GC-MS).[7] Despite the complementary strengths of these methods, there remains no standardized analytical protocol for combining GC-MS and LC-MS in VOC analysis at actual fire scenes.

This study aims to establish a comprehensive analytical strategy that applies both GC-MS and LC-MS to simultaneously capture volatile and semi-volatile compounds generated in fire environments. By proposing optimized pretreatment methods tailored to each instrument, this approach enables a more robust identification of fire-related chemicals and offers foundational data for chemical classification in forensic investigations, as well as for environmental toxicity and risk assessments.[8]

Experimental

A. Reagents and instruments

For gas sampling, an APK 1000 air sampler (KNR, Namyangju, Gyeonggi-do, Korea) and AirTox II sorbent tubes (KNR, Namyangju, Gyeonggi-do, Korea) were used[9] (Figure 1).

GC-MS analysis was conducted using a TD-GC/MSD

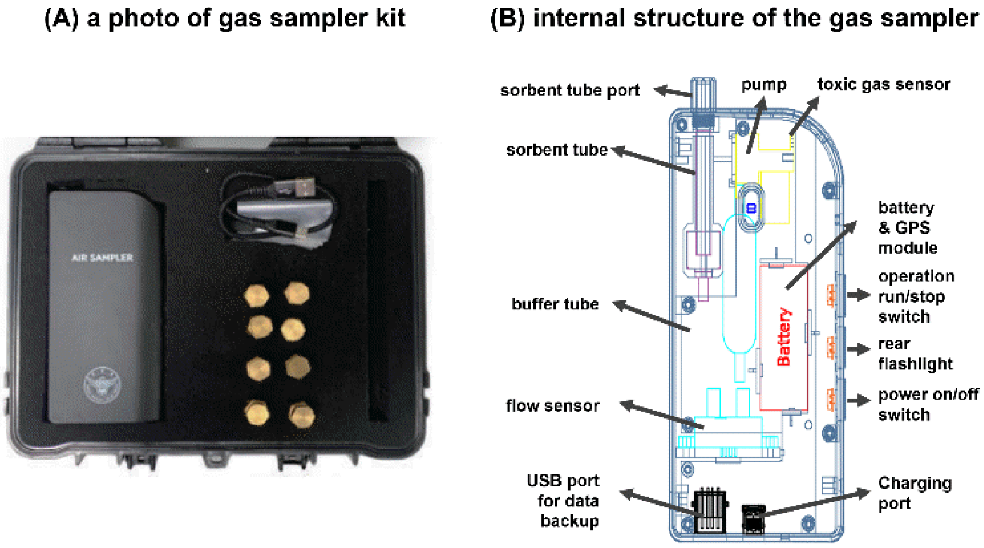

Figure 1. Photograph and internal structure of the gas sampler kit used for VOC collection at fire scenes.

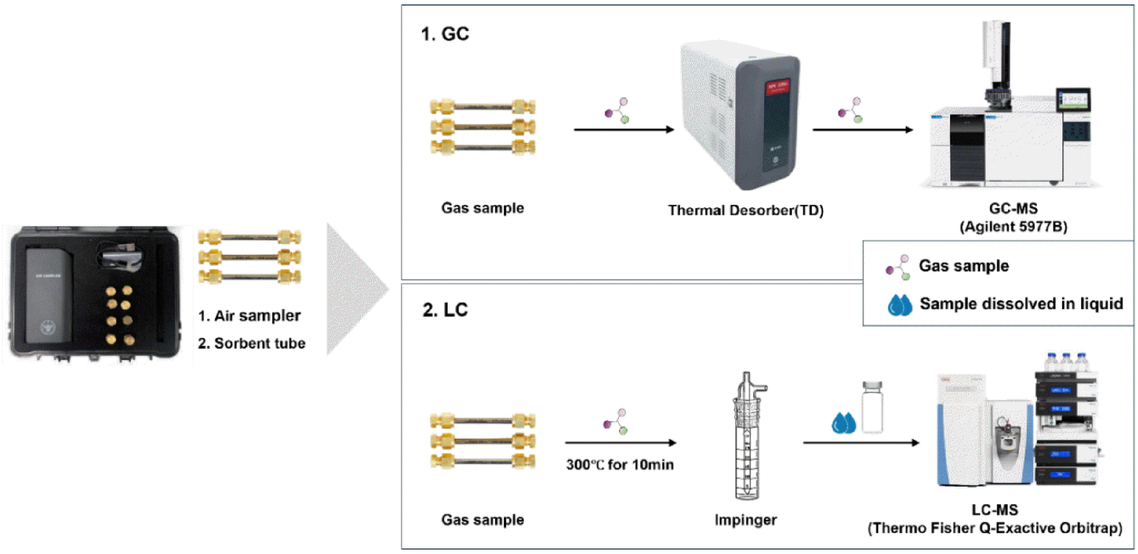

Figure 2. Integrated experimental workflow for fire scene VOC analysis using GC-MS and LC-MS

(Thermal Desorption-Gas Chromatography/Mass Selective Detector) system in combination with MassHunter software (version B.10.0, Agilent Technologies, Santa Clara, CA, USA). Compound identification was carried out using the NIST and Wiley spectral libraries.[10,11]

For LC-MS analysis, HPLC-grade acetonitrile (ACN) and 0.1% formic acid (FA) solution were used as solvents. Sample reconstitution and concentration were performed using a SpeedVac concentrator and an impinger system.

The analytical platform consisted of a UPLC-Exactive system (Thermo Scientific, Rockford, IL, USA) equipped with an Eclipse Plus C18 RRHD column (1.8 µm, 2.1 × 50 mm, Agilent Technologies, Santa Clara, CA, USA). Data processing was conducted using Compound Discoverer 3.3$^{\text{TM}}$ (Thermo Scientific, Rockford, IL, USA), and compound identification was based on the mzCloud (ddMS^2) spectral library (Figure 2).

B. Gas collection and analysis by GC-MS

Smoke generated from the combustion of wood was used as the target gas during fire simulation. The gas sampling device was placed within 1 meter of the combustion source, and the diffused smoke was collected for 20 minutes. For gas sampling, an APK 1000 air sampler and ten AirTox II sorbent tubes were used, with the flow rate set to 100 mL/min. Among the ten samples collected during the combustion experiment, Samples 1 and 8 were excluded from analysis due to insufficient gas volume. The remaining eight samples, No. 2–7 and 9–10 were sealed and stored at 4°C under refrigerated conditions. To evaluate the potential influence of background compounds originating from the equipment or sorbent tubes, a blank experiment was conducted under the same conditions prior to combustion. GC-MS analysis was performed using a TD-GC/MSD system, with thermal desorption carried out at 320°C for 10 minutes. Data acquisition and analysis were conducted using MassHunter software and compound identification was based on spectral matching against the NIST reference spectral library using the “Find by Chromatogram Deconvolution” algorithm. A match score threshold of 70 or higher was applied, and the scan range was set to *m/z* 35–500, enabling detection of VOCs from low to mid molecular weights.

C. Gas collection and analysis by LC-MS

To identify residual compounds in the sorbent tubes, twelve samples from the same combustion experiment were analyzed using a UPLC–Exactive system. During the sample pretreatment process, silicone tubing for impinger use was connected to both ends of each sorbent tube, and the tube outlet was immersed into a 15 mL conical tube containing 5 mL of ACN. Nitrogen gas was then purged at a flow rate of 50 mL/min for 10 minutes, followed by thermal extraction at 300°C for 10 minutes under the same flow conditions to release residual compounds from the sorbent material. From the extracted solution, 1 mL was taken and dried using a SpeedVac concentrator. The residue was then reconstituted in 100 µL of 0.1% FA. LC-MS analysis was performed using an Eclipse Plus C18 RRHD column. The mobile phases consisted of 0.1% FA in water (solvent A) and 0.1% FA in 80% ACN (solvent B), with a flow rate of 0.2 mL/min. The gradient conditions were as follows: 0–2 min, 2.5%; 2–11 min, 12%; 11–15 min, 28%; 15–22 min, 60%; 22–25 min, 96%; 25–25.1 min, 2.5%; 25.1–30 min, 2.5% of solvent B. Mass spectrometric analysis was conducted over an *m/z* range of 50–500. The acquired data were processed using Compound Discoverer 3.3 with the workflow “Untargeted Metabolomics with Statistics: Detect Unknowns with ID using online database and mzLogic. Compound identification was performed using the mzCloud (ddMS^2) spectral library, and similarity matching was applied to each compound using ddMS^2 spectra. According to the Metabolomics Society’s criteria, only compounds with a mass error < 10 ppm and an mzCloud score > 80 were retained and assigned Level 2 or Level 3 confidence. Redundant metabolites were filtered based on average area intensity.

Results and Discussion

A. GC-MS analysis results

A total of 12 common hazardous chemicals were

Table 1. Number of VOCs detected per sample by GC-MS and commonly identified hazardous compounds

No.	Sorbent tube No.	Number of hit compounds	Common hazardous chemicals (*n*=12)
1	B0430	180	Acetic acid, Furfural, Sulfurdioxide,(2-Aziridinylethyl)amine, Benzene,Toluene, Difluorodimethylsilane, 2-Propenal, Benzaldehyde, Nonanal, Octamethylcyclotetrasiloxane, 2-Methylnaphthalene
2	B0244	238	
3	B0262	168	
4	B0424	174	
5	B0352	69	
6	B0256	88	
7	B0607	75	
8	B0427	119	

detected across eight VOC sampling tube samples. The number of VOCs in each sample with a spectral match score of 70.0 or higher against the NIST library hereafter referred to as "hit compounds" is summarized below (Table 1).

B. LC-MS analysis results

Utilizing mzCloud spectral matching with a score threshold of ≥ 80 and a mass error limit of less than 10 ppm, a total of 52 compounds were annotated at level 2, while 1,351 compounds were identified at level 3. Following the exclusion of low-quality and non-reproducible signals, 950 unique compounds were retained to constitute the core dataset for subsequent LC–MS analysis. The number of detected compounds per sample ranged from 577 to 615, with 482 compounds consistently identified across all samples (Table 2). Notably, five compounds (benzaldehyde, furfural, nonanal, styrene, and ethylbenzene) were also detected via GC–MS. Among these, benzaldehyde, furfural, and nonanal were uniformly present in all analyzed samples (No. 2–7 and 9–10), whereas styrene and ethylbenzene were confined to samples 2–5 and 10 (Figure 3).

In this study, we employed both GC–MS and LC–MS to expand the detection range of VOCs released at fire scenes. Platform-specific sample preparation and collection parameters were optimized by taking into account the physicochemical properties of the analytes and differences in instrumental sensitivity. For GC–MS, the sampling flow rate and duration through sorbent tubes were initially based on literature-reported conditions and were subsequently adjusted to reflect the field environment. Consequently, VOCs were collected at a flow rate of 100 mL/min for 20 min, a condition that ensured sufficient analyte recovery while maintaining the operational efficiency required for field applications. For LC-MS analysis, an impinger-based method was applied to recover high-polarity and high-molecular-weight compounds. The impinger approach has previously been utilized in studies analyzing VOCs from indoor pollution sources generated by combustion, and in this study, it was also effectively applied for LC-MS analysis.[12]

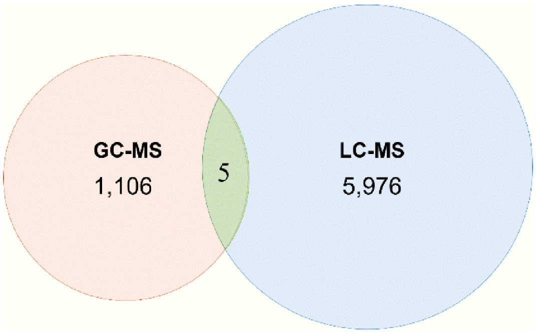

Figure 3. Comparison of the number of VOCs detected by GC-MS and LC-MS

We classified compounds with molecular weights below 200 Da as low-molecular-weight VOCs, whereas those 200 Da and above were considered relatively high-molecular-weight VOCs.[13] Additionally, the LC–MS scan range was intentionally restricted to below *m/z* 500 to ensure stable detection of polar and semi-volatile organic compounds. this limitation should be addressed in future studies by expanding the mass range. Consistent with this framework, GC–MS predominantly detected low-molecular-weight, highly volatile species such as acetic acid (60.5 Da), benzene (78.1 Da), and toluene (92.1 Da), whereas LC–MS identified comparatively higher-molecular-weight compounds including 1-tetradecylamine (213.4 Da) and 4-octylphenol (206.3 Da). These compounds, due to their higher polarity and molecular weight, are unsuitable for GC-MS but were detectable through LC-MS. Both compounds are classified as Substances of Very High Concern (SVHC) under the European Union's REACH regulation because of their bioaccumulative properties and potential endocrine-disrupting effects. This suggests that the consequences of fires may extend beyond immediate physical damage to long-term health and environmental risks.[14-16] In addition, five compounds were commonly detected by both

Table 2. Number of VOCs detected per sample by LC-MS and commonly identified hazardous compounds

No.	Sorbent tube No.	Number of hit compounds	Common hazardous chemicals (*n* = 12)
1	B0349	614	A total of 482 compounds were identified, including benzaldehyde, furfural, nonanal, styrene, ethylbenzene, benzothiazole, hexylamine, isophorone, 2-butoxyethanol, 1-tetradecylamine, *n*-butyl lactate, 2,2,6,6-tetramethyl-4-piperidinol, octadiene, 4-octylphenol
2	B0430	609	
3	B0244	615	
4	B0262	605	
5	B0424	594	
6	B0352	592	
7	B0256	585	
8	B0412	591	
9	B0607	599	
10	B0427	577	

GC-MS and LC-MS. These overlapping results enable cross-verification between analytical methods and can serve as a compound baseline, thereby enhancing the overall reliability of the analysis. From the standpoint of experimental design, the gas sampling methodology employed in this study was specifically developed to facilitate both GC–MS and LC–MS analyses from identical sorbent tube samples, thereby differentiating it from traditional VOC sampling techniques. Given that the primary objective was to broaden the qualitative detection spectrum of fire-related VOCs rather than to achieve absolute quantification, internal standards were deliberately omitted. Instead, all samples underwent uniform processing conditions, and blank controls were conducted to evaluate background interference, thereby ensuring dependable qualitative profiling. Nonetheless, the incorporation of isotopically labeled internal standards is recommended for future investigations focused on quantitative risk assessment to enhance analytical precision and reliability. In addition, while this approach enabled complementary detection of semi-volatile and polar compounds, the vacuum concentration step during LC–MS sample preparation may result in the loss of highly volatile analytes through solvent evaporation. Therefore, the results of this study should be interpreted with this potential limitation in mind. In conclusion, this study implemented an analytical strategy that enables the accurate and reliable detection of VOCs released at fire scenes, based on an integrated GC-MS and LC-MS analytical framework and sample preparation methods tailored to the characteristics of each instrument. Furthermore, the proposed strategy may serve as a reference for future research related to the assessment of hazardous substance exposure, the enhancement of reliability in forensic evidence interpretation, and the development of response measures for toxic substances. It may also provide foundational data for evaluating potential regulatory considerations or applications.

Conclusion

This study experimentally demonstrated that a combined analytical strategy utilizing GC-MS and LC-MS can effectively expand the detection range of organic compounds generated at fire scenes. By applying pretreatment methods optimized for each instrument and adopting a sequential analysis strategy using the same sorbent tubes, both analytical accuracy and operational efficiency were improved. This approach provides a practical foundation that considers both forensic reliability and field applicability. However, since the experiments were conducted under controlled laboratory conditions, there are limitations in fully reflecting the complex environmental variables of actual fire scenes. Future studies should address this by incorporating various combustion scenarios and real-world samples. In addition, quantitative evaluation and toxicological interpretation of key detected compounds, along with the development and potential standardization of analytical protocols, should be pursued. These efforts will contribute to multiple domains, including environmental and human exposure assessment following fires, toxic substance management, and the establishment of reliable forensic evidence systems.

Acknowledgments

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Conflicts of Interest

There are no conflicts to declare.

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