

Preliminary evaluation of a next-generation portable gas chromatograph mass spectrometer (GC-MS) for the on-site analysis of ignitable liquid residues

Alexander Visotin^{1,2} and Chris Lennard^{1,3*}

¹ National Centre for Forensic Studies, University of Canberra, Canberra, Australia

² GKA Investigations Group, Campbelltown, Australia

³ School of Science and Health, University of Western Sydney, Richmond, Australia

* Corresponding author. Email: c.lennard@uws.edu.au

Phone: +61 (0)2 4570 1739

Abstract

The detection of ignitable liquid residues (ILRs) at a fire scene can be valuable evidence to indicate if the fire may have been deliberately set. In this study, a commercially-available portable GC-MS, the TRIDION™-9, was evaluated for the purpose of on-site fire debris analysis. The instrument, which incorporates a low thermal mass capillary GC and a miniaturised toroidal ion trap mass spectrometer, is designed to use solid-phase microextraction (SPME) as the default sampling method.

Four ignitable liquids (petrol, mineral turpentine, kerosene and diesel) and seven substrates (nylon, polypropylene and wool carpet, rubber and foam underlay, untreated pine and polyurethane) were considered. After method optimisation, substrates were burned and spiked with 0.1 µL of ignitable liquid (mineral turpentine, kerosene, diesel fuel, and both neat and weathered unleaded petrol). Sampling was performed via passive headspace SPME adsorption over 3 min immediately followed by analysis using the TRIDION™-9 (analysis time <2 min). Field trials were performed on fire debris samples collected from an accelerated structural fire. The data from the portable GC-MS analyses permitted the detection and correct classification of ILRs in the majority of samples analysed. Overall, the TRIDION™-9 was found to be well-suited to the field-based analysis of fire debris samples as a preliminary screen to provide rapid presumptive results for fire investigators.

Keywords: fire investigation; arson; accelerants; portable instrumentation; hydrocarbons; presumptive screening.

Introduction

The presence of an accelerant at a fire scene may indicate that an act of arson has taken place as opposed to an accidental blaze. An accelerant may be any material used to facilitate ignition or contribute to the speed or spread of a fire¹. Arson, being the wilful or malicious burning of property, especially with criminal or fraudulent intent, is considered one of the easiest crimes to commit, yet one of the hardest to investigate². Ignitable liquids (ILs) are common accelerants as they are volatile and easy to ignite, with petrol (gasoline) being the most common due to its widespread availability³. If a liquid accelerant was used to propagate a fire, the analysis of debris collected from the scene relies on the fact that unburned ignitable liquid residues (ILRs) may still be present due to incomplete combustion. With this in mind, samples of burned debris from areas of interest are packed in hermetically-sealed containers (such as nylon or composite bags, or unlined metal tins) and submitted for laboratory analysis.

The isolation, detection and identification of ILRs in fire debris is a lengthy, time-consuming laboratory procedure. Forensic laboratories generally use a process consisting of sample

assessment, sample preparation (ILR extraction), instrumental analysis, data interpretation and reporting⁴. The isolation of ILRs from each sample can be the longest of these steps, depending on the method chosen. A common sampling method involves passive headspace adsorption onto activated charcoal. The adsorbed residues can then be isolated by solvent extraction, with the resulting solution typically analysed by gas chromatography mass spectrometry (GC-MS). The GC-MS results are then interpreted and compared to results from reference standards before a sample can be conclusively reported as containing an ignitable liquid. The timeframe from the collection of fire debris samples at the scene through to the reporting of laboratory results to the fire investigator can be significant and may be from weeks to months.

Unlike laboratory instrumentation, portable instruments can provide real-time, actionable data to assist investigators. Rapid presumptive results can help direct an investigation, long before the results from a full laboratory analysis are available. The need for accurate and reliable field-portable instruments in a forensic context has long been recognised. For example, diagnostic field tests can play an important role in terms of dealing with dissipating evidence⁵. This is a pertinent issue when analysing fire debris for ILRs. However, portable GC-MS technology was only making modest advancements at the turn of the century⁶. At that point in time, few field-portable GC-MS systems were truly portable. Most were vehicle-borne mobile laboratory setups or small ‘towable’ (or ‘transportable’) instruments rather than hand-portable devices. The development of portable GC-MS systems has been slow compared to laboratory-based instrumentation due to difficulties in satisfying performance requirements, funding limitations and technological feasibility⁷. However, the advent of low-thermal mass (LTM) GC systems and lightweight, high-performance batteries has had a significant impact on instrument portability and operational utility⁸. These innovations have legitimised commercial interest in field-portable GC-MS technology, encouraging the development of novel instruments for on-site applications. In addition to general requirements for diagnostic field tests, a portable GC-MS system has supplementary requirements that are relevant to the analysis of forensic specimens^{9–11}:

- Small size;
- Robust analytical ability;
- Limited power usage;
- Minimal consumables; and
- Rapid analysis and turnaround time.

A portable GC-MS system that meets these criteria is the TRIDION™-9, developed by Torion Technologies Incorporated (American Fork, Utah, USA; <http://torion.com>). The instrument, marketed as the world’s smallest person-portable GC-MS, weighs 14.5 kg and is completely self-contained (Figure 1). Features of the instrument include a LTM GC with high-speed temperature programming and a miniaturised toroidal ion trap mass spectrometer. The mass spectrometer has a mass spectral range of 45 to 500 amu, with better than unit mass resolution to 300 amu and nominal unit mass resolution to 500 amu. Samples are introduced into the instrument using a

novel SPME syringe (Figure 2). Data processing (i.e., peak deconvolution and compound identification) is performed by on-board software. The manufacturer claims that the TRIDION™-9 can perform up to 150 runs using a high-purity helium carrier gas canister, and can run for up to 2.5 hours using a rechargeable lithium ion battery. Results are displayed on a 5.7-inch colour LCD touchscreen and the instrument can be operated using its keypad or a stylus. Data can also be exported to a laptop for further analysis if required.

The instrument has been previously evaluated for the analysis of a number of target compounds including chemical warfare agents¹², solvents¹³ and contaminants in drinking water¹⁴. An early study in our laboratory produced encouraging results for the analysis of illicit drugs but some issues were encountered with the analysis of organic explosives¹⁵. When applied to the analysis of hydrocarbons, however, excellent results were achieved and this encouraged us to undertake further research to assess the instrument's application for the on-site analysis of fire debris samples. This is the subject of the study presented here and, to our knowledge, the instrument has not been previously evaluated for this application.

Materials and Methods

Instrument and Accessories

GC-MS analyses were performed on a TRIDION™-9 (Torion Technologies, Inc., Utah, USA) fitted with a 5 m MXT-5 capillary column (0.1 mm x 0.4 µm). Sample introduction was via a CUSTODION™ SPME syringe fitted with a 65 µm DVB/PDMS SPME fibre. Instrument calibration and performance checks were conducted as recommended by the manufacturer using a CALION™ Performance Validation (PV) 13-compound test mixture. Mass spectrometer settings were adjusted in accordance with the user manual and data analyses were performed using CHROMION® software version 1.1.1.6.

Ignitable liquids

Four ignitable liquids were selected for analysis: unleaded petrol, mineral turpentine, kerosene and diesel fuel. Petrol was sampled in both neat and weathered (evaporated) states, including weathering levels of 70%, 90% and 97% (w/w). For example, 70% weathered petrol was produced by evaporating down neat petrol until 30% of the starting weight remained. Neat petrol was sourced from a service station, while the other ignitable liquids were obtained from local retailers.

Method Optimisation

A standard method for the analysis of ignitable liquids was developed specifically for the TRIDION™-9 instrument. Due to the gas chromatograph's unusually short column and the use of an ion trap instead of a quadrupole, the typical GC-MS parameters published in the literature were not readily transferable. Default instrument methods supplied by the manufacturer were used as a starting point for the optimisation process, which involved an assessment of the parameters listed in Table 1. Parameters that were not varied, as recommended by the instrument manufacturer, included the injection port temperature (270°C), the transfer line temperature (250°C) and the SPME desorption time (10 sec).

The test sample employed for initial method optimisation consisted of 0.1 µL **neat** unleaded petrol deposited on a section of absorbent paper that was then sealed in a 1-litre new, unlined paint tin (supplied by NCI Packaging, Victoria, Australia). After equilibration, a hole was pierced in the centre of the sample tin lid using a hammer and nail. The SPME syringe was inserted into the hole and the fibre exposed to the headspace of the sample for the specified sampling time. SPME sampling was conducted at room temperature (20–22°C). After sampling, adhesive tape was applied to the hole in the tin lid to preserve the sample. GC-MS analysis was performed by insertion of the SPME syringe into the injector of the TRIDION™-9 and exposing the SPME fibre for the default desorption time as instructed by the instrument's software. Each of the remaining ILs was analysed in a similar fashion and parameters adjusted as required before finalisation of the method.

At the beginning of each sample analysis period, a specific procedure was followed that included running system blanks, SPME fibre blanks and a CALION™ PV check. The PV runs served as an autocalibration process for the instrument and to alert the user to system problems. Instrument parameters including filament emission, signal resolution and electromagnetic detector response were tested during the PV procedure. Peak resolution and mass spectral quality were automatically evaluated via the PV process to determine which spectrometer components required tuning. System maintenance was performed as required, including cleaning the ion trap and replacing consumable parts.

GC-MS Data Analysis

Compounds considered to be diagnostically relevant, such as those listed in ASTM Standard Test Method E1618-11¹⁶, were chosen as target compounds (Table 2). These were added to the TRIDION™-9's on-board library using the target library editor in the CHROMION software. Target compound identifications were based on retention times and full mass spectra.

Interpretation of the GC-MS data generated by the TRIDION™-9 was based on ASTM Standard Test Method E1618-11¹⁶ and Australian Standard 5239-2011¹⁷. Total ion chromatogram (TIC) patterns were examined first, including sample elution ranges, peak distribution, and the

presence of target compounds. Second, ion profiles were extracted for all major compound types of interest. The most abundant ion profiles were compared to those extracted from reference chromatograms and peak ratios were compared. In samples where pyrolysis product interference made the visualisation of target compounds problematic, analyses were conducted using extracted ion profiles (EIPs). Individual compounds were identified according to their retention times and mass spectra.

Simulated fire debris samples

Blank substrate materials were sourced from various locations. Nylon carpet, polypropylene carpet and wool carpet swatches, as well as rubber and foam underlay, were collected from a retail flooring outlet. A section of untreated pine wood as well as a children's sofa (polyurethane) were obtained from a waste recycling centre. Substrates were cut to size and then burned using a butane/propane portable camp burner. Substrates were burned as evenly as possible across their surface area until significant charring was visible on all sides. At this point, a 0.1 μL volume of ignitable liquid was deposited onto a section of filter paper. The filter paper was immediately packaged with the burned substrate in a 1-litre new, unlined paint tin to simulate a fire debris sample. Samples of burned and unburned substrates without an ignitable liquid were also prepared in a similar fashion. SPME sampling and GC-MS analyses were performed on all samples as described previously.

Analysis of the burned and spiked samples resulted in a finding of either (1) presumptive identification of an IL, (2) presumptive indication of the presence of an IL, or (3) no IL detected. Samples that contained the majority of expected target compounds and generated EIPs that closely represented those from reference ILs were assessed as a presumptive identification (good correlation). Samples where some target compounds were missing and where compound peak ratios were not entirely consistent with reference ILs were assessed as a presumptive indication only (partial correlation). Samples where few target compounds were present and where chromatograms were inconsistent with reference ILs were assessed as not containing an ILR.

Real fire debris samples

To supplement the spiked samples described above, real fire debris samples were generated as part of a research exercise hosted by Fire and Rescue NSW. This exercise involved the controlled burn of a single-storey house and two adjacent structures in Gilgandra, NSW. In the first part of the study, simulated house floor surfaces were burned. The floors consisted of a shipping pallet supporting the floor materials, flanked by two sections of plasterboard. The surfaces included treated timber, synthetic carpet, linoleum, wool carpet, ceramic tiles and a wooden floating floor. After ignition, the substrates were allowed to burn until self-extinguishment, with all but one accelerated with an IL (petrol). Floor samples were then

collected approximately 24 h later, having been exposed to ambient daytime temperatures of around 16°C. In the second part of the study, two samples were retrieved from the controlled burn of a bedsit structure, where a petrol accelerant had been poured in two locations. Sample collection took place approximately 1 h after extinguishment. Three more samples were collected from the controlled burn of the living room area of the main structure, where kerosene had been sprayed as an accelerant. Fire debris samples were collected from each location after the location had been screened by a trained accelerant detection canine (ADC) to see if an alert was elicited. Each debris sample was sealed in a 1-litre new, unlined paint tin. SPME sampling and TRIDION™-9 analyses, using the methods described above, were then performed on-site. Note that, for the samples collected from the bedsit and living room fires, the instrument operator was unaware of the nature of any IL that may have been employed as an accelerant but they were aware of any alerts from the ADC.

In-Field Instrument Evaluation

During the research exercise conducted in Gilgandra, the general performance of the instrument in the field was evaluated. This included an assessment of how robust the instrument was for on-site applications. Factors such as the longevity of the instrument's battery power supply and the number of analyses per disposable helium canister were examined. An informal evaluation of the instrument's usability was also conducted in terms of assessing how easy it was to move around, set up, perform analyses and interpret results.

Results

Method Optimisation

Whilst longer SPME sampling times generally provided better results, with more intense compound peaks in the chromatogram, a modest sampling time of 180 sec (3 min) was chosen as the best balance between rapidity and reliability. A column ramp rate of 3°C/sec resulted in poor chromatographic separation but a fast analysis time (Figure 3). While a ramp rate of 1°C/sec provided the best peak resolution, 2°C/sec was ultimately selected as a compromise between analysis time and chromatographic separation, and because 2°C/sec was the ramp rate required for the instrument's PV method. The column start temperature and hold time had minimal impact on the results so this was set at 50°C with a zero hold time. Diesel, the heaviest petroleum distillate analysed, was used to determine the final column temperature and hold time. Alkanes up to octadecane (C18) could be visualised in the diesel fuel chromatograms. To ensure that all detectable target compounds eluted, a final column temperature of 270°C and a hold time of 5 sec were selected. Several injection types were investigated, ranging from splitless injections to basic 10:1 and 50:1 splits and combination splits. Combination splits involved using both split ratios (10:1 and 50:1, one after another) and a delayed split (turning the split on several seconds

after the injection). Splitless injections were not appropriate as this produced a large amount of background noise in the chromatogram and poor peak resolution. Basic splits were more effective. However, results were significantly improved with the use of a delayed mixed split, with improved peak resolution and sensitivity being achieved with this mode. The parameters selected for the optimised method are summarised in Table 3. Examples of chromatograms recorded under these conditions are provided in Figure 4 and Figure 5.

Simulated fire debris samples

Analysis of the blank, unburned substrates showed little volatile content with the exception of the untreated pine sample, which produced a small number of intense peaks in the chromatogram. The two largest peaks in this chromatogram represented α -pinene and β -pinene, with smaller peaks identified as camphene and limonene. The same compounds were identified in the chromatogram from the burned sample of pine. Burning of the other substrates produced a range of pyrolysis products that included, depending on the substrate, toluene, ethylbenzene and styrene.

The results from the burned and spiked samples are summarised in Table 4. In most cases, for the majority of the substrates tested, the IL was detected and correctly classified. However, the burned pine was problematic as the chromatograms largely represented terpene peaks that were associated with the substrate itself; IL target compounds, when present, were very low in concentration. This interfered with ILR detection and recognisable EIPs could not be generated for many of these samples. The false negative results may have been due to the preferential adsorption of terpenes on the SPME fibre, with little remaining capacity to capture the IL target compounds, and/or competitive adsorption between the SPME fibre and the charcoal on the burned pine substrate.

The detection of diesel in the burned nylon carpet sample and the detection of mineral turpentine in the burned polyurethane sample also produced negative results, largely due to the prevalence of interfering pyrolysis products. With all of the remaining samples, there was either a presumptive indication or a presumptive identification of the corresponding IL. As an example, the results for burned polyurethane spiked with 0.1 μ L kerosene are shown in Figure 6, demonstrating the use of EIPs to remove interfering peaks due to pyrolysis products. Figure 7 compares the results obtained from burned nylon carpet (no IL), 70% weathered unleaded petrol, and burned nylon carpet spiked with 0.1 μ L 70% weathered unleaded petrol.

Real fire debris samples

For the first phase, petrol was correctly identified in the fire debris samples from the linoleum, wool carpet, ceramic tiles and wooden floor burns. For the synthetic carpet sample, pyrolysis

product interference was apparent, with styrene being the most prominent peak in the chromatogram. While a small number of target compounds for petrol were detected, these were not present in the ratios expected and a negative finding for ILRs was reported. This was a false negative result as petrol had been used to accelerate this burn. Note, however, that the ADC did not alert at this location prior to sample collection and analysis. For the treated timber sample, the GC-MS data indicated the presence of terpenes as well as some of the target compounds expected for petrol, leading to a conclusion that petrol residues may have been present. This was a false positive indication as no accelerant had been used in this instance and the target compounds detected were probably from the substrate itself. Again, the ADC had not alerted at this location and, in addition, subsequent laboratory analysis on a bench-top GC-MS failed to indicate the presence of an IL in the sample.

In phase two, the samples from the bedsit structure were correctly reported to contain petrol residues and the samples from the living room of the main structure were correctly reported to contain kerosene samples. The TRIDION™-9 results from the real fire samples are summarised in Table 5. In all cases where there was the presumptive identification of an IL, the ADC had given a positive alert prior to sample analysis.

In-Field Instrument Evaluation

During the research burn exercise in Gilgandra, the TRIDION™-9 was operated from the boot of a car (Figure 8). This proved to be an efficient mobile laboratory setup, allowing for samples to be brought to the instrument from the fire scene while keeping the instrument, accessories and samples in a relatively clean environment. Note that such a location should only be employed if the analysis of a SPME background sample confirms that the area is free of any detectable volatile organic compounds. In general, the TRIDION™-9 performed similarly in the field to how it performed in the laboratory during the analysis of the simulated (burned and spiked) fire debris samples.

SPME fibre blanks run after each sample typically showed no signs of ghost peaks or other contamination artefacts. Highly concentrated samples did overload the SPME fibre occasionally, leaving analytes on the fibre after the first injection. One or two additional injections were sufficient to clean the fibre in these instances.

During the field exercise, the battery power was depleted after approximately 2.5 h, corresponding to around 25 sample analyses (which included system start-up, PV checks, SPME blanks, and sample runs) and in agreement with the manufacturer's claims. A subsequent check under laboratory conditions, with the continuous running of blank samples, gave similar results. A full helium cylinder outlasted two fully-charged battery packs (around 50 analyses), with approximately 50% of the canister's capacity remaining. This suggests that a single canister of

carrier gas would be sufficient for around 100 runs, compared to the manufacturer's indication that the capacity was sufficient for up to 150 analyses.

Some hardware/software issues were encountered, including low-battery false alarms and the system sometimes losing its time and date settings. Otherwise, the instrument was generally reliable and could be easily packed in its carry case, moved around, and unpacked for operation. No adverse effects on the analyses were observed when the instrument was transported in this fashion. The system is typically ready for operation in around 5 min from a cold start, with a PV check being the first recommended analysis.

The instrument's user interface was relatively easy to use and navigate. Menu items on the LCD interface were all that were required to run analyses in the field, although connection to a laptop provided additional functionality including more advanced data analyses. The instrument was generally easier to control with the touch-screen stylus rather than using fingers to navigate the user interface. However, despite its general ease of use, deployment of the TRIDION™-9 for this application requires experience with the use of GC-MS for fire debris analysis. While technical officers and laboratory technicians may be able to use the instrument and understand data output, their ability to interpret the data to determine whether or not an ILR is present will be limited by their experience in forensic chemistry. A portable GC-MS of this type should therefore only be deployed with a forensic chemist who has been specifically trained in the operation of the instrument and in the interpretation of GC-MS data from fire debris samples containing ILRs.

General Maintenance and Troubleshooting

General maintenance operations were found to be fairly simple to perform and described clearly enough in the instrument's user manual for most operators to follow. Disassembly of the instrument, while unlikely to be required in the field, can be accomplished by anyone with a basic understanding of how the instrument works. However, disassembly of internal componentry such as the mass analyser or the electron multiplier detector should only be performed under controlled laboratory conditions. Such cleaning procedures should only be undertaken by personnel familiar with the instrument and specifically trained to perform the required maintenance. Reassembly and proper alignment of the ion trap, for instance, is a precise process that can have varying degrees of success. In this study, the ion trap sometimes required reassembly and realignment several times after a clean in order to attain acceptable mass resolution. The ion trap required disassembly and cleaning approximately every 500–600 analyses, with the need determined by a loss of mass resolution and/or a loss of mass accuracy.

General troubleshooting of the instrument also requires some experience. Advice is given by the automated tune wizard on the instrument to correct for PV failures; however, the tune wizard

was found to sometimes give ambiguous or unhelpful advice. Space charge effects, a common pitfall of ion trap technology, were problematic during some sample analyses. With the TRIDION™-9, space charge issues are symptomatic of a dirty ion trap or an excessively high filament voltage. Both result in more ions being pushed into the ion trap and overloading it, resulting in higher ion values being recorded by the detector. In most cases, cleaning the ion trap resolved these issues. However, some space charge effects occurred intermittently even when the ion trap was clean, and seemed to be pronounced in some sample types more than others.

Loss of high mass resolution occurred sporadically throughout the research project. In the first instance, it occurred due to a bug in the instrument software that discounted all ions with a mass above 250 amu. This was resolved with a software upgrade. Other losses of resolution that were encountered generally resolved themselves after several days or after an instrument reset. Towards the end of the study, a persistent loss of high mass resolution was noted. Ions above 300 amu did not resolve optimally, though resolution was still acceptable for routine analyses. Despite these periodic issues, mass resolution was otherwise excellent, with unit mass resolution or better for masses up to 300 amu. Operationally, sporadic losses in high mass resolution are not a concern for the detection of ILRs but could be an issue for other applications such as the analysis of illicit drugs or explosives.

Discussion

The small ignitable liquid volume (0.1 µL) employed in this study for spiking the simulated fire debris samples was based on the minimum requirement for detection as specified by ASTM Standard Practices E2154-01¹⁸. It is stated in this document that “[SPME is] capable of isolating small quantities of ignitable liquid residues from a sample, that is, a 0.1 µL spike of gasoline on a cellulose wipe inside of a 1-gal can is detectable”. The detection and correct classification of ignitable liquids at this volume confirmed the acceptable performance of the SPME sampling method and the associated GC-MS analyses. Using only 0.1 µL of IL on burned substrates – some with prominent interfering pyrolysis products – deliberately challenged the analytical capabilities of the TRIDION-9 as well as the data analysis capabilities of the associated software. The positive identification of residues at such small volumes in burned debris was an impressive outcome given the speed of analysis and the portability of the TRIDION™-9. Note that larger spiking volumes were tested during the early stages of this study, producing excellent results and an increased level of confidence in the identifications.

The 3-min SPME sampling period and 2-min GC-MS analysis time could conservatively permit the on-site analysis of up to 12 samples per hour (5 min per sample). If two SPME syringes are available then sampling can be conducted on one item while the GC-MS analysis is being undertaken on another. It is advisable, however, to run frequent SPME blanks to ensure that there is no carry-over of material between analyses. Periodic PV checks should also be

performed to ensure that the instrument is operating correctly. It is recommended that this is done at instrument start-up and at least daily if the instrument is running continuously.

No amount of analytical power can substitute for a properly collected sample. Thus, a detection method should be used at the scene to locate samples potentially containing ILRs that can then be analysed using the TRIDION™-9. The evaluation of the instrument in Gilgandra showed that it can be effectively deployed alongside ADCs in the field. The canine can locate a sampling site and the handler can pass this information on to the scene examiner, who can then collect a debris sample and have this analysed by the TRIDION™-9 operator. Timely feedback can then be provided to the scene examiner regarding ILR results and to the canine handler regarding the canine's performance.

It is our view that, given the limitations of on-site analyses (compared to analyses under controlled laboratory conditions), results from the TRIDION™-9 – or from other portable GC-MS systems of a similar nature – cannot be considered as conclusive evidence for the presence of ILRs in fire debris. Independent laboratory confirmation on a conventional bench-top GC-MS is still required. Nevertheless, the instrument is capable of producing a rapid result that, for positive samples, is a strong indication for the presence of a particular IL. From the receipt of a sample, the operator can generate a result in around 5 min that can be immediately communicated to the fire investigator.

Conclusions

The detection abilities of the TRIDION™-9 for four ignitable liquids on different substrate matrices were investigated during this study. ILRs were presumptively identified in 38 of 49 simulated samples. The presence of ILRs was presumptively indicated in four samples, and only seven samples gave negative results. The ability of the TRIDION™-9 to detect volumes as low as 0.1 µL of ignitable liquid on burned substrate matrices was notable. The detection and identification of ILRs in 9 of 11 real fire debris samples further suggests that the TRIDION™-9 would be a useful tool in operational casework. The instrument performed well in a field setting, with the rechargeable battery pack providing power for approximately 2.5 hours in accordance with indications from the manufacturer. In addition, each disposable helium canister should permit around 100 analyses.

A trained and experienced operator is required to perform maintenance on the system if performance is unsatisfactory and when regular servicing is necessary. Otherwise, any forensic chemist with a knowledge of GC-MS can be trained in the instrument's operation. Some issues with the instrument are persistent, such as the sporadic loss of high mass resolution and space charge issues; however, to a skilled operator, these effects make little difference to the data analysis process and the results achieved.

A limitation of the study was that an extensive comparison against results from a bench-top GC-MS was not undertaken. However, a small number of comparisons did confirm that similar results were obtained. More thorough comparisons will be performed in follow-up operational trials where samples analysed on-site with the TRIDION™-9 will be systematically analysed using conventional lab-based methods.

Despite the preliminary nature of this research, the TRIDION™-9 portable GC-MS shows significant potential for the on-site analysis of fire debris samples. Such analyses can serve a dual purpose at the fire scene. Firstly, it can be a presumptive testing tool used to screen fire debris samples on-site, acting as a level of quality control between the scene and the laboratory and ensuring that only the more relevant samples are submitted for full laboratory analysis. Secondly, the TRIDION™-9 is able to provide actionable forensic intelligence to fire investigators at the scene with respect to the rapid detection and identification of ILRs in collected samples. This is congruent with the current push in the forensic science community towards intelligence-led policing and may assist fire investigators in testing origin and cause hypotheses while still at the scene.

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Table 1. Parameters assessed as part of the method optimisation process.

Parameter	Test values
SPME sampling time	10, 30, 60, 120, 180, 300 sec
Starting column temperature	30, 40, 50°C
Starting temperature hold time	0, 10, 20, 30, 60 sec
Temperature ramp rate	1, 2, 3°C sec ⁻¹
Final column temperature	255, 270, 300°C
Final temperature hold time	0, 5, 10, 20, 30, 60 sec
Split ratio	Splitless, 10:1, 50:1, mixed split, delayed split

Table 2. Target compounds selected for the characterisation of the chosen ignitable liquids

No.	Compound	No.	Compound
1	toluene	18	2-methylnaphthalene
2	ethylbenzene	19	1-methylnaphthalene
3	m-/p-xylene	20	1,3-dimethylnaphthalene
4	o-xylene	21	2,3-dimethylnaphthalene
5	propylbenzene	22	octane
6	m-/p-ethyltoluene	23	nonane
7	1,3,5-trimethylbenzene	24	decane
8	o-ethyltoluene	25	undecane
9	1,2,4-trimethylbenzene	26	dodecane
10	1,2,3-trimethylbenzene	27	tridecane
11	indane	28	tetradecane
12	1,2,4,5-tetramethylbenzene	29	pentadecane
13	1,2,3,5-tetramethylbenzene	30	hexadecane
14	4-methylindane	31	heptadecane
15	5-methylindane	32	octadecane
16	4,7-dimethylindane	33	trans-decalin
17	naphthalene		

Table 3. Parameters employed in the final optimised method.

Parameter	Value
SPME sampling time	180 sec (3 min)
Starting column temperature	50°C
Starting temperature hold time	0 sec
Temperature ramp rate	2°C/sec
Final column temperature	270°C
Final temperature hold time	5 sec
Split ratio	Splitless (2 sec) 10:1 split (10 sec) 50:1 split (8 sec)
Total GC run time	115 sec (1.9 min)

Table 4. Portable GC-MS results from the simulated fire debris samples (✓✓ = presumptive identification of IL; ✓ = presumptive indication of IL; – = nil IL detected).

Ignitable liquid (0.1 µL) spiked on burned substrate							
Substrate	Unweathered petrol	70% weathered petrol	90% weathered petrol	97% weathered petrol	Mineral turpentine	Kerosene	Diesel
Nylon carpet	✓✓	✓✓	✓	✓✓	–	✓✓	✓
Polypropylene carpet	✓✓	✓✓	✓✓	✓✓	✓✓	✓✓	✓✓
Wool carpet	✓✓	✓✓	✓✓	✓✓	✓✓	✓✓	✓✓
Rubber underlay	✓✓	✓✓	✓✓	✓✓	✓✓	✓✓	✓✓
Foam underlay	✓✓	✓✓	✓✓	✓✓	✓✓	✓✓	✓✓
Untreated pine wood	–	–	–	✓✓	✓	–	–
Polyurethane	✓✓	✓	✓✓	✓✓	✓✓	✓✓	–

Table 5. Portable GC-MS results from the real fire debris samples (✓✓ = presumptive identification of IL; ✓ = presumptive indication of IL; – = nil IL detected).

		Accelerant deployed	ILR detected
Simulated floor surfaces	Treated timber	Nil	✓ (petrol)
	Synthetic carpet	Petrol	–
	Linoleum	Petrol	✓✓
	Wool carpet	Petrol	✓✓
	Ceramic tiles	Petrol	✓✓
	Wooden floating floor	Petrol	✓✓
Bedsit	Cushion	Petrol	✓✓
	Chest of drawers	Petrol	✓✓
Living room	Armchair	Kerosene	✓✓
	Flooring	Kerosene	✓✓
	Couch	Kerosene	✓✓

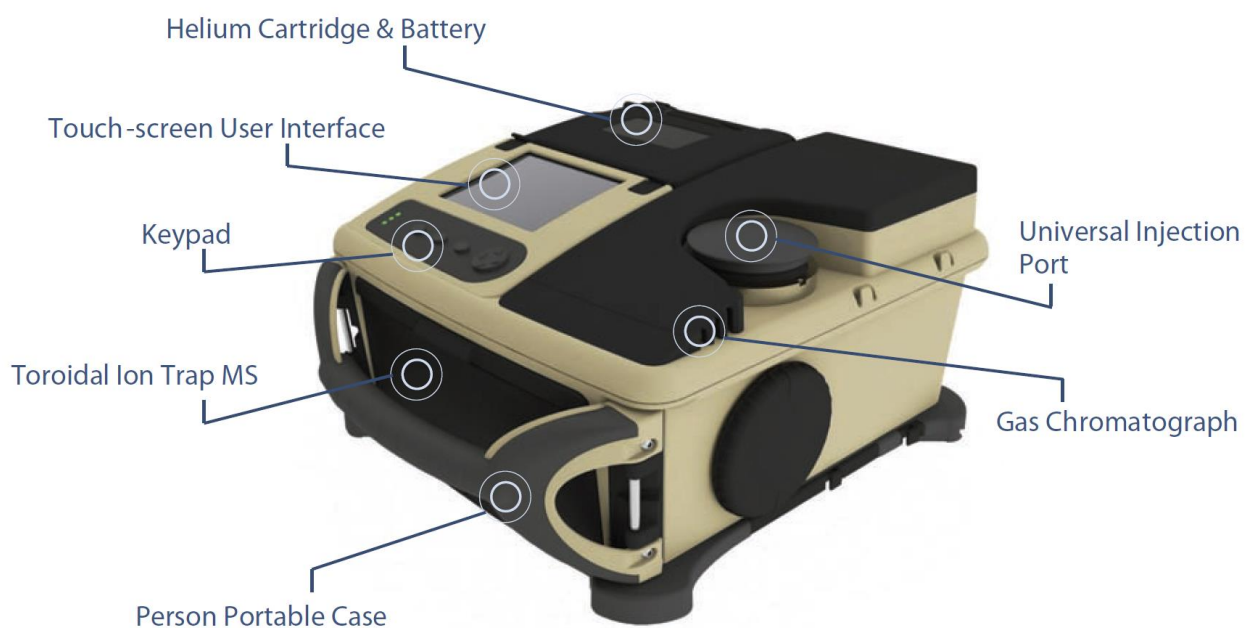


Figure 1. The TRIDION™-9 person-portable GC-MS. The approximate dimensions of the instrument are 38 x 39 x 23 cm. Image courtesy of Torion Technologies Incorporated (American Fork, Utah, USA; <http://torion.com>).



Figure 2. The CUSTODION™ SPME syringe used as the default sample introduction method with the TRIDION™-9 instrument. Image courtesy of Torion Technologies Incorporated (American Fork, Utah, USA; <http://torion.com>).

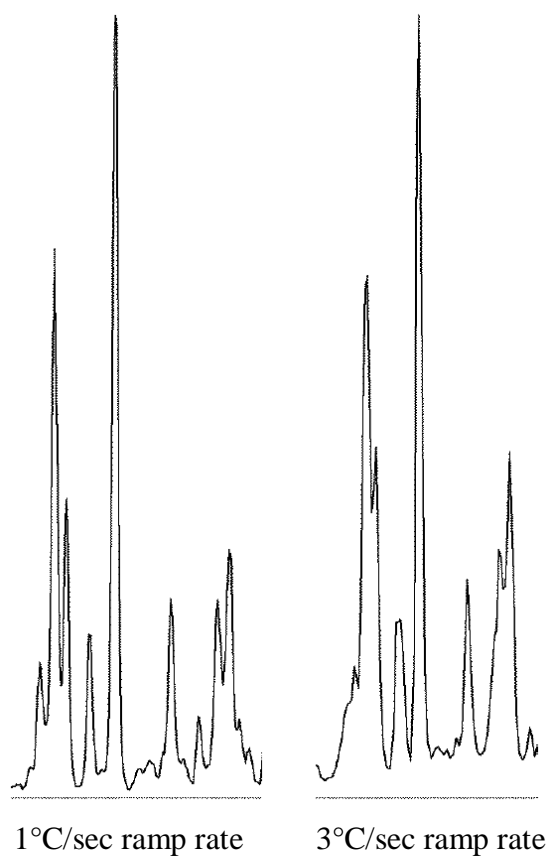


Figure 3. Comparison of the chromatographic separation achieved for two different column ramp rates. The two chromatograms represent the same group of compound peaks from a neat petrol sample.

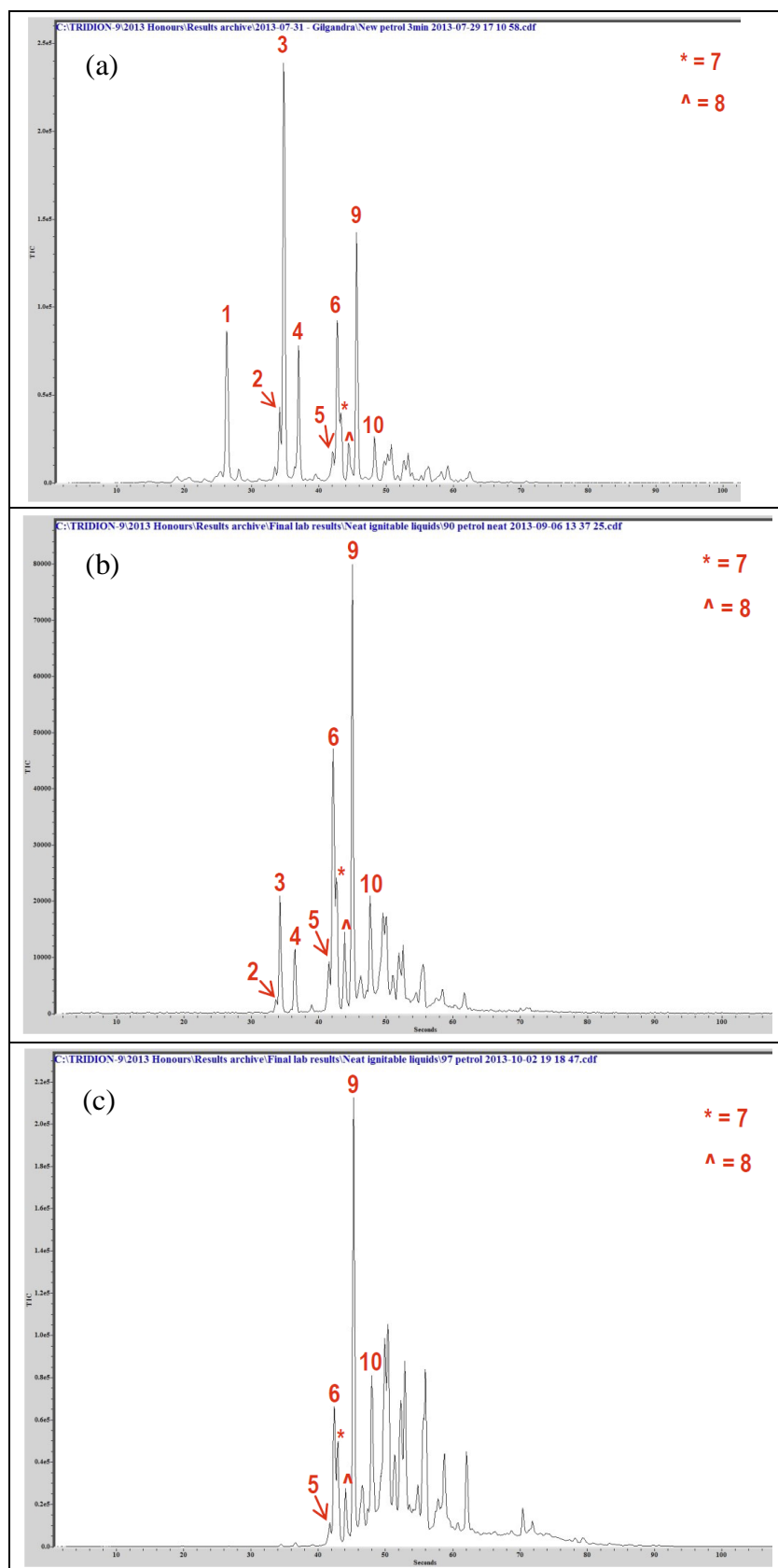


Figure 4. Chromatograms for (a) neat, (b) 90% weathered, and (c) 97% weathered unleaded petrol. (Numbers represent target compounds as listed in Table 2.)

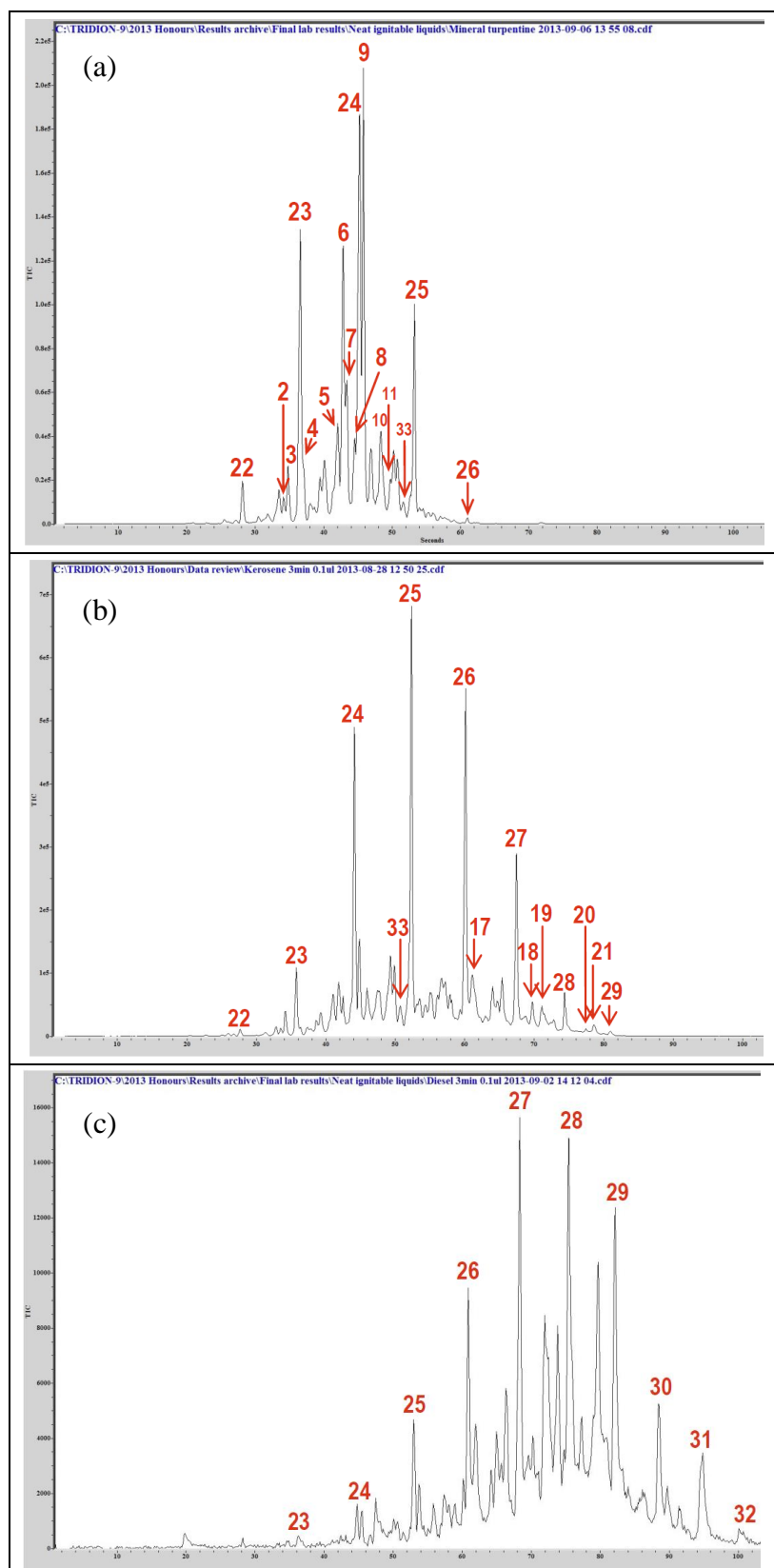


Figure 5. Chromatograms for (a) mineral turpentine, (b) kerosene, and (c) diesel fuel. (Numbers represent target compounds as listed in Table 2.)

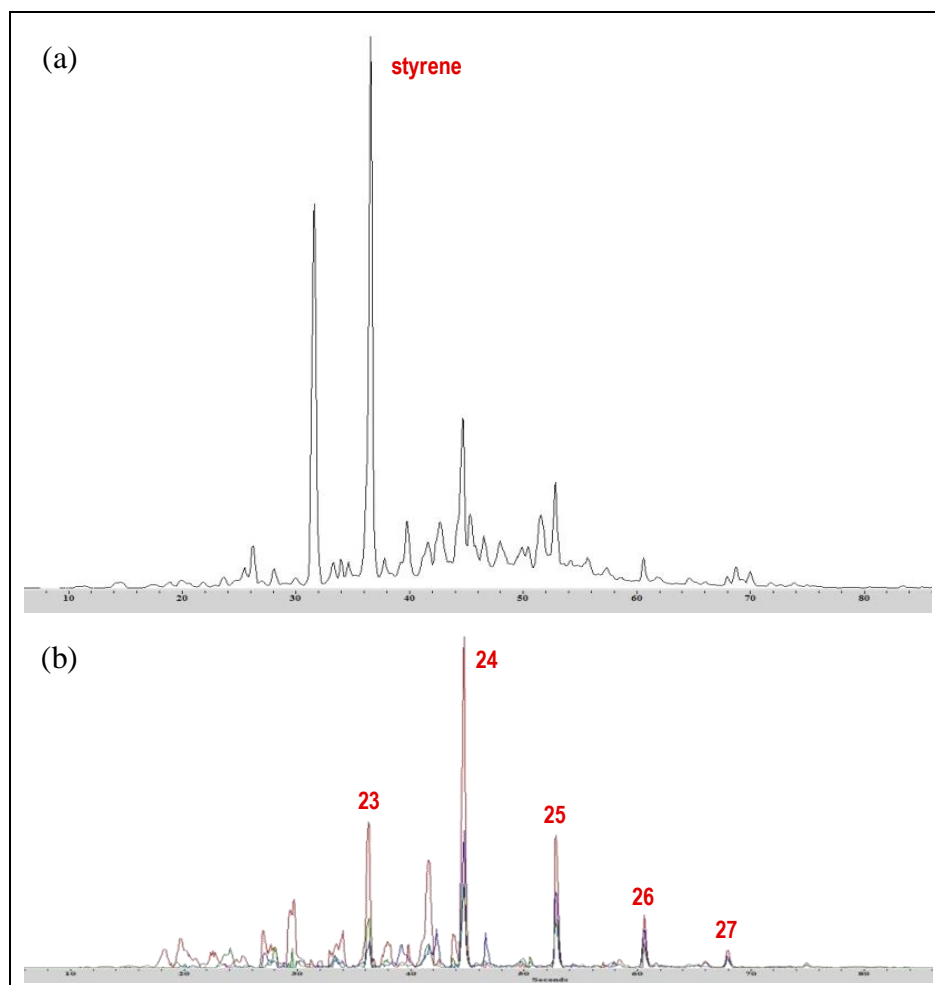


Figure 6. Portable GC-MS results for burned polyurethane spiked with 0.1 μL kerosene: (a) total ion chromatogram showing styrene as a prominent pyrolysis product; (b) extracted ion profile for alkanes (57, 71 and 85 m/z). (Numbers represent target compounds as listed in Table 2.)

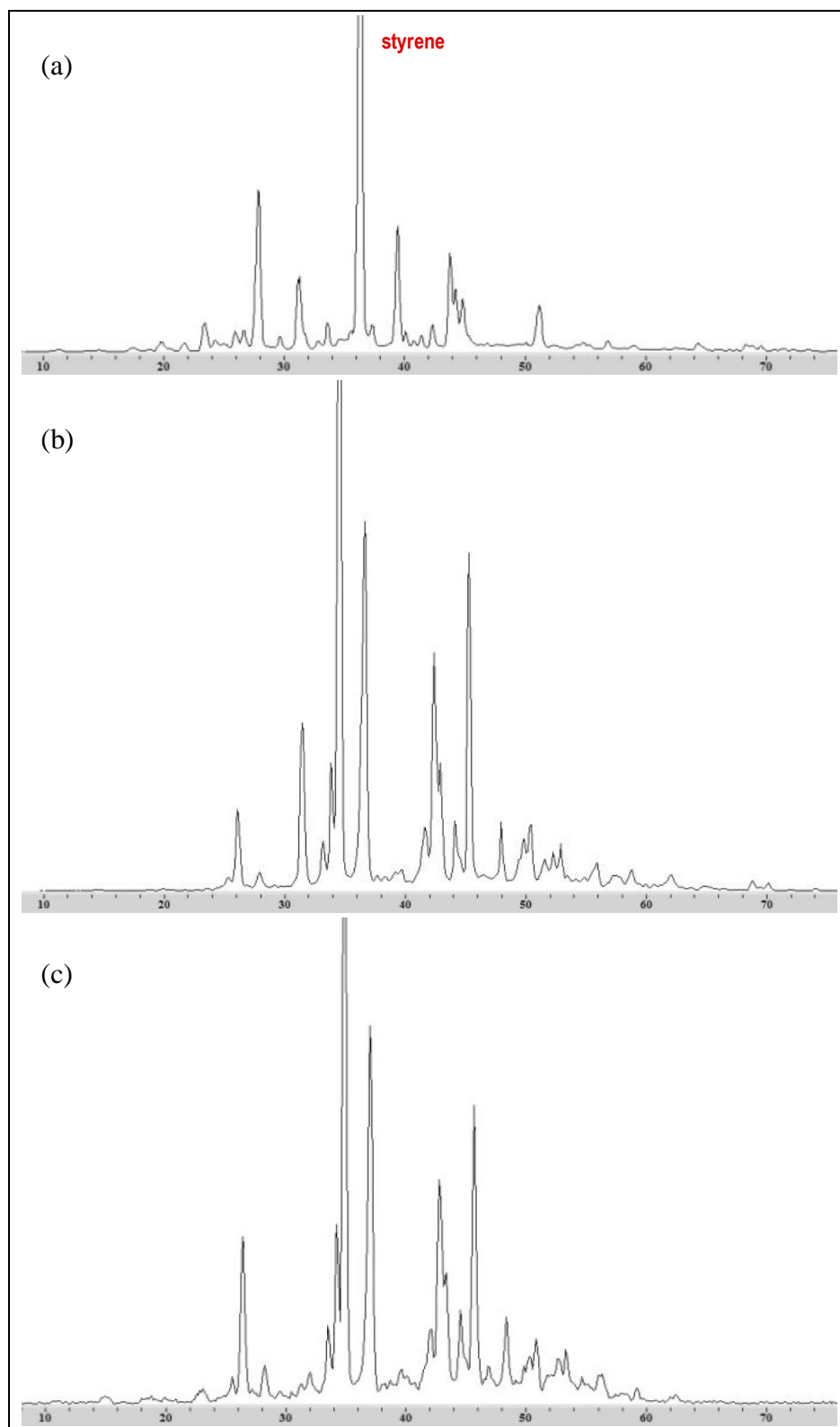


Figure 7. Portable GC-MS results for: (a) burned nylon carpet; (b) 70% weathered unleaded petrol; (c) burned nylon carpet spiked with 0.1 µL 70% weathered unleaded petrol.



Figure 8. The mobile laboratory setup employed during the Gilgandra research exercise.