

Developments in Analysis of Fire Debris Residues

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Abstract

This review gives a brief overview of the recent developments in the field of analysis of petroleum products in fire debris residues by different spectroscopic and chromatographic techniques for forensic purpose. The review covers different aspects of analysis such as the types of accelerants, substrates involved, isolation procedures, column and mobile phase used, and subsequent detection in tabular form. This paper covers detection of petroleum products such as petrol, kerosene and diesel in various types of samples recovered in arson cases.

Keywords: Fire Debris Residues; Inflammable Liquids; Petroleum Products; Solid Phase Microextraction; Gas Chromatography-Mass Spectrometry.

Introduction

Any chemical substance used to initiate or increase the intensity or speed of spread of fire is known as fuel. It is used to intentionally start or spread fire. Not all inflammable liquids found at a fire scene are accelerants and conversely, not all accelerants used to commit arson are liquids [1,2].

Accelerant may be a solid, liquid or in some cases gas. Liquid fire accelerants include highly inflammable and volatile liquids petroleum products, solvents etc. Petroleum products such as petrol, kerosene and diesel are more frequently used than other inflammable liquids such as alcohols, paints, lacquer thinners, ether and industrial solvents [1].

Arson is one of the most difficult crimes to investigate because of its destructive nature. The crime itself destroys the physical evidence at its origin. Most of the evidences are destroy in burning process and rest are destroy during fire extinguish

process. Arson is the one crime that destroys, rather than creates evidence as it progresses [3].

Flammable liquids are not frequently present in case of unintentional fires and unexplained presence of these liquids thus strongly indicates a fire of suspicious origin. Detection and identification of these accelerants are therefore helpful in determining origin and cause of fire [4].

The crime of arson falls under the Indian Penal Code (1860) section 435, 436 and 438. Arson is considered as violent crime affecting the public safety and peace [5].

Petroleum products (petrol, kerosene and diesel) are frequently used to commit arson due to their easy availability, simple handling, storage and cost effectiveness. Petrol is highly inflammable and volatile substance. Kerosene is less volatile than petrol. It is available as a subsidized domestic cooking fuel and is often misused as a fire accelerant in arson and bride burning cases [1].

Detection of these accelerants is thus of vital importance. Partially burnt exhibits including clothes, carpet, wood, soil, hairs, paper, concrete recovered from body of victim, accused and crime scene may be referred to forensic science laboratories for the detection and characterisation of petroleum residues. Sample should be stored with utmost care and its loss and contamination should be avoided during transfer to laboratory and following analysis as it affects the outcome of analysis and also raises the question about the integrity of sample in court. Standard sampling procedure should be followed with the proper documentation in order to uphold sample integrity and chain of custody [3,6].

Different kinds of containers can be used for this purpose. Sample containers may vary from bags to jars and metal cans and each of them has their own merits and demerits. Containers used to store the fire debris sample should be clean, inert, and durable and does not itself provide a source of cross-contamination. It is observed that many types of plastic bags contain interfering compounds [6].

Tontarski Jr. advocates that polyethylene containers should be avoided to collect the fire debris samples due to significant accelerant loss and sample contamination. Therefore, containers of unknown quality should be avoided and only "certified containers" should be used [7].

Fire debris may contain trace amounts of inflammable substances. Many classical physical and chemical methods are used for the detection and identification of accelerants in fire debris residues. But these methods are time-consuming, less sensitive and not reliable. Therefore, sophisticated instrumental analytical techniques are frequently used to analyse trace amounts of accelerants in fire debris residues.

Thus on the basis of frequent use of petroleum products in arson cases, it seems to be necessary to summarize recent spectroscopic and chromatographic procedures used to identify them in fire debris residues. So, we select the detection of petrol, kerosene and diesel in various complex matrices of forensic interest by different conventional and modern instrumental techniques. The aim of this compilation is to give an overview on analysis of petroleum products (including extraction procedure, separation and identification by different techniques) in different kinds of matrices. Table 1 reflects the characteristics of different GC and GC-MS methods for analysis of accelerants in fire debris samples.

Analysis of Petroleum Products

Development of analytical methods that are capable of detecting trace amounts of petroleum products in fire debris samples has become increasingly important in the field of forensic science. Routine analyses rely on the visual comparison of chromatogram of sample with chromatogram of standard petroleum product. Most of reviews provide information regarding the different sample preparation methods used for the analysis of the accelerants in fire debris residues [8-10].

There is a need for a review providing recent information on their analysis in different arson debris samples. Therefore, we compile different analytical methods (including extraction) used for analysis of petroleum products in fire debris samples. Petroleum products such as petrol, kerosene and diesel are frequently encountered in arson cases and in cases of suicidal or homicidal burning associated with bride burning in dowry disputes. These accelerants are present in traces in partially burnt exhibits such as mattress, wood, plastic, paper, carpet, clothes etc. Techniques used for isolation and concentration of accelerants from different substrates have changed from time to time. In 1950's and 1960's, steam distillation, vacuum distillation and solvent extraction procedures were used to extract accelerants from substrates and are considered as conventional extraction procedures [11,12].

In early 1970's, headspace extraction procedures were introduced in fire debris analysis and are considered as modern extraction procedures [6,11].

These headspace extraction procedures include direct (heated) headspace analysis, static headspace analysis such as carbon strip method, solid phase micro-extraction (SPME) and dynamic headspace analysis (purge and trap analysis). Various screening methods and instrumental techniques can be used for the extraction and detection of accelerant residues. These methods are discussed as follows:

Spectroscopic Methods

Spectroscopic methods are based on measurement of signal generated by the interaction of electromagnetic radiations with matter. These methods provide both qualitative and quantitative information about the analyte. However, due to the impurities and complex nature of petroleum products and matrices received in arson cases, these methods are not frequently used to establish the identity of analyte. These methods can be used as a screening

method at the initial stage of analysis.

Adams described the different procedures (I. and II.) used for the extraction of small amounts of accelerants from arson debris depending on boiling product (low or high). In procedure I., air was pumped through the system at rate of about 1l/min for 2 to 3 mins. Sample was collected in jar and concentrated by heating with a 300W reflector flood bulb. Sample may be recovered as a mixture of solid and liquid. Procedure II. involves the washing of debris with carbon tetrachloride. These washings were collected, combined and concentrated by evaporation at room temperature. Infrared spectroscopy was used to pinpoint the identity of accelerants. He concluded that these procedures were efficient and rapid for the extraction of minute quantities of accelerants from arson debris [13].

Bryce et al. analysed fire debris samples using nuclear magnetic resonance spectroscopy. Samples were subjected to steam distillation in order to extract accelerants from samples and an aliquot amount of distillate was placed in 2ml sample vials. Tetramethylsilane was used as an internal standard. Deuterated chloroform was used as a solvent. They observed that technique is independent of interfering substrate due to its low sensitivity. They also observed that different solvents have no significant effect on spectrum. As little as 0.25ml of sample is sufficient for analysis. They concluded that nuclear magnetic resonance spectroscopy complements the present analytical techniques and could provide a powerful tool for use in fire debris analysis [14].

Meal analysed the fire debris samples by using second derivative ultraviolet spectroscopy. Samples were extracted with cyclohexane and extracts were scanned in the range of 245-320nm. Quartz cells of 1cm path length were used. He observed a unique and easily recognizable second derivative UV spectrum of different accelerants including thinners and spirits and concluded that second derivative ultraviolet spectroscopic method is rapid, sensitive, simple and cost effective for analysis of fire debris and independent of matrix interference [15].

Mc Curdy et al. developed a method for the analysis of arson accelerants in fire debris by using vapour phase ultraviolet spectroscopy. They compared the present method with conventional GC-FID and GC-MS method and found that vapour phase ultraviolet spectroscopy is rapid, inexpensive and sufficiently sensitive to be of use for the analysis of arson accelerants. 10cm path length quartz cell was used. Spectra were recorded over the wavelength range 230-280nm. 1 μ l of sample is sufficient to record vapour phase UV spectra. They concluded that

method could be used as alternative to currently existing techniques for analysis of arson accelerants [16].

Liu et al. developed a portable and cost effective cataluminescence based vapour sensitive sensor array for rapid detection and discrimination of flammable liquid vapours. They used linear discriminant analysis and hierarchical cluster analysis to discriminate fingerprints of flammable liquid vapours. Photomultiplier tubes were used to detect the cataluminescence signals. Heating voltage for sensor array was maintained at 5.5V. They observed that water vapours did not cause significant interference in detection process. They also observed that different analytes could be 'fingerprinted' by their unique patterns of cataluminescence intensities. They noted that sensor array reduces the interference caused by complex combustion background. They concluded that method is useful to discriminate flammable liquid vapours of forensic interest [17].

Chromatographic Methods

Chromatographic methods are used to separate the components of complex mixture. These methods are widely used due to their simplicity, rapidity, sensitivity and cost-effectiveness. These methods cannot provide the definite identity of analyte due to their non-specificity. But coupling of these methods with appropriate spectroscopic method not only enhances the sensitivity but also the selectivity of technique.

Thin Layer Chromatography/High Performance Thin Layer Chromatography (TLC/HPTLC) Methods

Baggi et al. presented a simple and effective thin layer chromatographic method for the detection and characterization of petroleum residues extracted from different substrates such as cloth, paper, wood and leather. 100ml of distillate was collected and extracted with benzene. The benzene fractions were collected, combined and evaporated to dryness at room temperature. n-heptane saturated with 3% v/v formaldehyde was used as mobile phase in TLC. Concentrated sulfuric acid or anisaldehyde was used as chromogenic reagent. Thin layer chromatographic plates coated with silica gel G impregnated with 2% w/w sodium carbonate and silica gel GF 254 were used. They observed that as little as 0.02ml of petroleum residue could be detected and characterized by this method. They also observed that sodium carbonate impregnated plates were more effective than other plates in separation of components of analytes [18].

Dhole et al. developed a method for the detection of petroleum accelerant residues on partially burnt objects in arson or bride burning cases. The method involves the conversion of aromatic hydrocarbons such as naphthalene and alkyl naphthalenes into corresponding phenols or naphthols which were further detected by thin layer chromatography or high performance thin layer chromatography after spraying with diazotized p-nitroaniline reagent followed by aqueous ethanolic sodium hydroxide solution. A mixture of chloroform : xylene (3:1) was used as a solvent system. They observed that petroleum products could be easily differentiated with respect to total number of spots, their colour, position, shape and relative distribution and concluded that although the present method is time consuming and laborious yet it is more sensitive and selective than conventional thin layer chromatographic methods [19].

Malve et al. analysed the dye component of different fake and adulterated petrol samples (genuine petrol, fake petrol, adulterated green petrol) by employing high performance thin layer chromatography. The samples were evaporated on water bath (60-70°C) and residue was dissolve in 0.2ml of benzene. 20µl of sample was spotted on plate and a mixture of hexane: ethyl acetate (19.5:0.5) was used as solvent system. They also studied distillation characteristics of these samples and observed that fake and genuine samples have different temperatures at 90% recovery points. They concluded that distillation characteristics along with dye analysis can be used to confirm the purity and adulteration of petrol samples [20].

Gas Chromatography-Flame Ionization Detection (GC-FID) Methods

Gas chromatography was first applied to fire debris analysis in 1960 [21]. Prior to that, ignitable liquid residues were analysed by different techniques [13,22-25].

Flame ionization detector (FID) is most popular detector used with GC and it is considered as a universal detector for GC. Number of organic samples can be analysed by FID and most GC analyses are performed using FID. Its response is not influence by change in the flow rate of mobile phase and it work as a standard detector for the analysis of hydrocarbons from last three decades due to its high sensitivity, low noise and large linear response range [26].

Lloyd analysed petroleum products of high relative molecular mass by capillary gas

chromatography and observed that paraffin wax was mostly resolved while other samples form the characteristic unresolved envelope. He concluded that present method can be used to distinguish different types of petroleum products of high relative molecular mass and visual inspection of chromatograms are sufficient to made such discrimination [27].

Twibell et al. compared the different methods used for the extraction of accelerants from fire debris. They observed that capillary columns offers higher sensitivity than packed columns due to their greater resolution. They concluded that adsorption wire procedure is most sensitive while hot headspace method is least sensitive whereas steam distillation has intermediate sensitivity [28].

Nowicki and Strock compared different extraction techniques used for analysis of fire debris samples. They also optimized the different parameters of extraction procedures. Peak area of 1,2,4-trimethylbenzene was selected to study the efficiency of extraction method. They observed that efficiency of headspace method can be optimized by heating 10ml of sample at 120°C for 20mins. They concluded that charcoal adsorption/elution technique is more efficient than headspace and solvent extraction [29].

Frenkel et al. compared the headspace and adsorption tube extraction technique for the analysis of arson residues. They also compared the two desorption methods (thermal desorption such as pyrolysis and solvent extraction) used with adsorption tube technique. They also studied the effect of water on analysis of arson residues. They suggested that Zeolite 4A (0.4nm pores, 2g/1ml of water) molecular sieves could be used to eliminate the interference caused by water. They observed that solvent extraction method was as sensitive as thermal desorption (such as pyrolysis) but cracking was observed in later. They concluded that adsorption tube method is more sensitive, efficient and useful than headspace method [30].

Reeve et al. compared charcoal adsorption and direct headspace injection techniques to isolate accelerants from fire debris and concluded that direct headspace injection technique is simple, sensitive and require minimum sample preparation [31].

Mann compared and discriminate different gasoline samples (neat and evaporated) on the basis of the relative peak intensities. He suggested that boiling point range, relative concentration of major versus minor components, aliphatic and aromatic hydrocarbon content and presence of additives are useful parameters to distinguish different classes of

ignitable liquids. He concluded that present comparison method cannot be used to formulate positive conclusion while it is an ideal method to formulate negative conclusion and used for screening of samples at initial stage of investigation [32].

Waters and Palmer advocated the utility of passive headspace concentration technique in multiple analysis of fire debris and concluded that present concentration technique is non destructive, fast and less susceptible to contamination [33].

Almirall et al. developed a method for the extraction and identification of accelerants from aqueous samples of fire debris by using SPME-GC-FID. They also compared this method with solvent extraction procedure using hexane as an extracting phase. They observed the higher sensitivity of SPME as compared to solvent extraction procedure for light petroleum distillate and gasoline while solvent extraction of diesel did not produce identifiable chromatograms. They concluded that SPME technique is more sensitive, less laborious, rapid, solventless technique for the extraction of ignitable liquid from aqueous samples than conventional solvent extraction method [34].

Furton et al. developed a novel method for the analysis of gasoline from fire debris by using SPME. They also compared this method with passive adsorption elution method and concluded that SPME technique is faster, cost-effective, efficient, simpler and sensitive than conventional passive adsorption elution method and it provides reproducible multiple analyses without the need of any organic solvent [35].

Newman et al. studied the effects of different parameters such as adsorption time, adsorption temperature, strip size and concentration of sample on the adsorption of accelerants. They observed that diesel and kerosene products were difficult to discriminate at temperature less than 60°C.

However, higher temperature (>90°C) was not recommended due to pyrolysis and decomposition of substrates. 8mm × 8mm strip size and heating at 50-70°C for 16-24hrs is recommended to produce efficient results. They observed that amount of displacement of lighter components in sample rises with increase in temperature, time, concentration and size of strip [36].

Almirall et al. developed a method for detection and identification of flammable liquid residues on human hand by using SPME-GC-FID. Accelerant was deposited on the hand of a subject and it was then covered with a nylon bag. PDMS coated SPME fibre

was inserted into bag and it was exposed to headspace over the hand for 15 mins. Fibre was then inserted into the injection port of gas chromatograph. They concluded that present method is sensitive, simple, inexpensive, fast to detect and identify flammable liquid residue from human skin and can be used in conjunction with an accelerant detection canine team at crime scene [37].

Coulson et al. studied the effect of compressed air foam on detection of hydrocarbon fuels in fire debris samples and concluded that air foam did not cause significant interferences in detection and identification of hydrocarbon fuels [38].

Bodle and Hardy developed a SPME based method for the analysis of petroleum based accelerants. SIMCA and PCA techniques were used to identify and classify the accelerants. The accuracy of classification by SIMCA models for previous and current ASTM system was 98.5% and 97.2% respectively. They concluded that SIMCA is an effective class predictor of accelerants [39].

Darrer et al. compared the efficiency of different mediums (PVC, PE and Latex gloves, humidified filter paper) to collect gasoline from hands. They also evaluated the persistency of petrol on hands using PVC gloves. They observed the massive evaporation of gasoline within 30mins of its deposition on hands and concluded that PVC gloves are effective and efficient medium for collection of gasoline from hands due to low background noise or reduced amount of volatile compounds detected from it [40].

Sanagi et al. developed a method for the analysis of accelerants in fire debris samples by using headspace single drop microextraction (HS-SDME). 10ml of sample solution was placed in 15ml headspace vial and continuously stirred. 2.5µl of benzyl alcohol was withdrawn into a microsyringe and this microsyringe was then inserted into headspace and suspended over sample solution. Benzyl alcohol was then suspended on the tip of syringe needle and drop was withdrawn into syringe after 20 mins of extraction. Then extract was directly injected into injection port of GC. They observed no interferences due to burnt matrix. They concluded that HS-SDME-GC-FID is a rapid, sensitive and simple method and can be used as an excellent alternative method for the analysis of accelerants in fire debris samples [41].

Gas Chromatography-Photo Ionization Detection (GC-PID) Method

This detector measures the current generating from

photoionization of analyte by ultraviolet radiation at a suitable electrode. Its high sensitivity for aromatic hydrocarbons, higher selectivity and large linear response range enhances its utility in analysis of complex hydrocarbon mixtures. Thus the combination of FID and photoionization detection for the analysis of complex samples is more useful and advantageous [26].

Higgins et al. compared the extraction efficiencies of two heating techniques, i.e.; convection heating and microwave oven heating. Samples were placed in a polyester bag and metal paint can for microwave oven and convection heating respectively. Accelerants were isolated from carpet material by convection as well as microwave oven heating procedures and these isolated accelerant vapours were adsorbed on activated charcoal sample tube which were further washed with 2ml of carbon disulfide in order to extract accelerants. 1 μ l of extract was injected into the injection port of GC. Photoionization detector was used due to its high sensitivity. They observed that microwave oven heating procedure is more efficient and heat the samples faster than convection heating. They concluded that conventional convection oven can be replaced with microwave oven for sample heating in order to reduce analysis time and to enhance the sensitivity of accelerant vapour collection [42].

Gas Chromatography-Mass Spectrometry (GC-MS) Methods

Gas chromatography-mass spectrometry is an extremely versatile instrumental technique for the analysis of wide range of volatile compounds. In GC-MS, a gas chromatograph (GC) is attached to a mass spectrometer (MS) via a suitable interface. GC-MS is a hyphenated technique in which two techniques based on completely different principles are coupled together. In GC-MS, GC separates the components of sample while MS identify them. Compounds are separated on the basis of their relative affinity with stationary phase of column. Components eluting from the chromatographic column are then introduced to the mass spectrometer via a specialized interface. Coupling of MS with GC not only detects the analytes but also provide the pinpoint identification of analytes. Therefore MS provides a powerful detection tool in combination with GC [26].

Smith analysed arson debris and standard accelerants (neat and evaporated) by GC-MS used in extracted ion profile mode, i.e., mass chromatography. He observed that mass chromatography can eliminate the interference

caused by combustion and pyrolysis products of matrices. Method provides the rapid detection of specific compounds even in the presence of intense background interferences and concluded that mass chromatography is a valuable tool for examining arson debris samples [43].

Tranthim-Fryer described a simple, sensitive and cost effective carbon wire adsorption/solvent extraction technique for the analysis of accelerants and volatile organic compounds in arson debris. He observed that water vapors did not cause any interference in detection and identification of accelerants and concluded that present method allows further examination of samples at later date (if required) and can be suited to those laboratories without a thermal desorption unit or pyrolyzer [44].

Keto and Wineman identified petroleum based accelerants in fire debris by GC-MS. Target compound chromatograms (TCC) of samples were compared with TCC's of their corresponding standards to establish the identity of sample. They observed that target compound patterns for fresh and weathered gasoline, medium petroleum distillates and heavy petroleum distillates were specific and could be used for their identification in high background arson debris samples. They also observed that pyrolysis products have different TCC's from petroleum products. They concluded that target compound analysis is a simple, easy and useful approach for the identification of residual petroleum products in fire debris [45].

Coulombe analysed fire debris samples and different products (low volatility residue from gasoline, evaporated diesel fuel, evaporated gasoline from Molotov cocktail and creosote) by GC-MS and concluded that diphenyldisulfides and its homologues could be considered as chemical markers of weathered gasoline since these components were not present in creosote and other petroleum derivatives. However, their absence cannot eliminate the presence of gasoline [46].

Frontela et al. compared two extraction procedures used to extract accelerants from arson debris by GC-MS in specific mass ion mode and concluded that although adsorption/elution method is faster than distillation but later has higher degree of efficiency [47].

Steffen and Pawliszyn developed SPME method for the detection of liquid accelerants from arson suspected fire debris by using GC-FID and GC-Ion Trap MS. They observed that interferences from arson samples or fire debris could be distinguished from trace amounts of accelerants by using GC-

Table 1: Characteristics of the GC methods for analysis of accelerants in fire debris samples

S. No.	Analyte/s	Matrix	Extraction Technique/s	Column (Dimensions)	Carrier gas (flow rate)	Detect or	Injector & Detector Temp.	m/z range	References
1	Petrol, kerosene, diesel	NR	Dynamic adsorption/ elution	DB-1 (30m × 0.25mm × 0.25µm) BP-5 (32m × 0.32mm × 0.5µm)	N ₂ (1ml/min) He (1ml/min)	FID, MS	265°C, 280°C	45-500	[16]
2	Paraffin wax, liquid paraffin, gear oil, light oil, lubricating oil, engine oil	NR	NR	OV-1 glass capillary (50m × 0.25mm)	H ₂ (4ml/min)	FID	280°C	NA	[27]
3	Petrol, kerosene, diesel	Hardwood charcoal	Hot headspace, steam distillation, adsorption wire technique	OV 1 (25m) 15% Carbowax 20M on chromosorb W packed column (3m × 6.35mm)	N ₂ (10psi=inlet pressure) N ₂ (60ml/min)	FID	NR	NA	[28]
4	Petrol	Carpet padding	Solvent extraction, steam distillation, direct headspace, sorbent trap/thermal desorption	Supelcoport stainless steel (12' × 1/8'')	N ₂ (20ml/min)	FID	230°C, 230°C	NA	[29]
5	Petrol, kerosene, diesel	Filter paper	Headspace, Dynamic adsorption/elution	OV-101 Stainless steel (6m × 3.175mm)	NR	FID	NR	NA	[30]
6	Petrol, kerosene, diesel	Paper	Headspace, Passive adsorption/ elution	DB-5 (30m × 0.32mm × 1µm)	H ₂ (70cm/sec)	FID	250°C	NA	[31]
7	Petrol, MPD, HPD	NR	Passive adsorption/ elution	SPB-1 (30m × 0.32mm × 0.25µm)	He (2ml/min)	FID	250°C, 290°C	NA	[33]
8	Petrol, diesel, lighter fluid	Water	Solvent extraction, SPME	HP-1 (30m × 0.2mm × 0.25µm)	He (1ml/min)	FID	220°C, 300°C	NA	[34]

9	Petrol	Burned pine, wood, plastic	SPME, Passive adsorption/elution	HP-1 (30m × 0.25mm × 0.25µm)	1ml/min	FID	220°C, 300°C	NA	[35]
10	Petrol, kerosene, diesel	Wood, kimwipes	Passive adsorption/elution	DB-1 (12m × 0.25mm × 0.25µm)	He (25.6cm/sec)	FID	275°C, 280°C	NA	[36]
11	Petrol, diesel, charcoal lighter fluid	Human hand	SPME	DB-5MS (30m × 0.25mm × 0.25µm)	He (1ml/min)	FID	250°C, 310°C	NA	[37]
12	Petrol, kerosene, diesel, white and methylated spirits, mineral turpentine, isopropanol	Tissue paper, soil, unburnt carpet and wood, burnt carpet and wood	Passive adsorption/elution	DB-1 (25m) Carbowax (30m)	He (30ml/min)	FID	For DCM extracts (260°C, 260°C) For water extracts (150°C, 150°C)	NA	[38]
13	130 accelerants including petrol, kerosene, diesel	NR	SPME	PETROCOL DH 50.2 (50m × 0.2mm × 0.5µm)	N ₂ (1ml/min)	FID	270°C	NA	[39]
14	Petrol	Human hand	Passive adsorption/elution	DB-5 (30m × 0.32mm × 1.0µm)	He (2ml/min)	FID	250°C, 280°C	NA	[40]
15	Petrol, diesel, kerosene	Fabric curtain	Headspace single drop microextraction	Ultra-1 (25m × 0.20mm × 0.1µm)	He (1.5ml/min)	FID	NR	NA	[41]
16	Petrol, dodecane	Carpet	Passive adsorption/elution	Stainless steel (3.65m × 3.18mm)	N ₂ (30ml/min)	FID	NR	NA	[42]
17	Petrol, kerosene, diesel	Tissue paper	Passive adsorption/elution	BP-1 (25m × 0.25mm × 0.25µm)	He (Head pressure= 120kPa and 35kPa)	FID, MS	NR	NR	[44]
18	Petrol, MPD, HPD	Wood, tile, carpet	Passive adsorption/elution	DB-1 (60m × 0.32mm × 0.25µm)	He (2ml/min)	MS	260°C	50-200	[45]

19	Petrol	NR	Steam distillation, Passive adsorption/ elution	HP-1 (25m × 0.2mm × 0.33µm)	He (0.5- 1ml/min)	MS	250°C	40-500	[47]
20	Petrol, barbecue lighter fluid	Wood, carpet	SPME	DB-5 (30m × 0.25mm × 0.25µm)	NR	FID, MS	275°C, 300°C	50-260	[48]
21	Petrol, diesel	Filter paper	Passive adsorption/ elution	HP-1 (25m × 0.2mm × 0.5µm)	He (100kPa)	MS	NR	33-300	[49]
22	Petrol	Cloth, paper towel	Passive adsorption/ elution	Rtx-1 (30m × 0.25mm × 0.25µm)	0.68ml/min	MS	NR	10-400	[50]
23	51 accelerants including Petrol, kerosene, diesel	Wood, carpet	Passive adsorption/ elution	Supleco (25m × 0.2mm × 0.5µm)	He (40ml/min)	MS	NR	NR	[51]
24	Diesel	Wood, chipboard, plastic, carpet	Passive adsorption/ thermal desorption	Elite-1 (30m × 0.25mm × 1.0µm)	He	MS	NR	NR	[52]
25	Petrol	Car carpet	Passive adsorption/ elution	DB-5MS (30m × 0.25mm × 0.25µm)	He (1.7ml/min)	MS	250°C	40-200	[53]
26	Petrol, lubricant, fuel oils	Water	Passive adsorption/ elution	Elite (1.30m)	NR	MS	NR	35-350	[54]
27	Petrol	Wood, paper, carpet	SPME	HP-5MS (30m × 0.25mm × 0.25µm)	He (1ml/min)	MS	280°C	40-400	[56]
28	Petrol	NR	SPE	HP-5MS (30m × 0.25mm × 0.25µm)	He (1.2ml/min)	MS	280°C	30-350	[57]

29	Petrol, diesel	Carpet (nylon, polyester, wool), wallpaper, synthetic floor	Passive adsorption/ elution	SBP-5 (30m × 0.25mm × 0.25µm)	He (1ml/min)	MS	275°C	37-400	[58]
30	Petrol	Wood	Passive adsorption/ elution	DB-1 (60m × 0.25mm × 1.0µm)	He	MS	NIR	29-200	[59]
31	Petrol	Carpet	Passive adsorption/ elution	HP-5MS (30m × 0.25mm × 0.25µm)	He (1.2ml/min)	MS	280°C	30-350	[60]
32	Petrol, kerosene, diesel, extraction solvent, universal diluter	Carpet, wood, unpainted chipboard	Passive adsorption/ thermal desorption	Elite-1 (30m × 0.25mm × 1.0µm)	He	MS	NIR	NR	[61]
33	Petrol, diesel, C ₁ -C ₄ alcohols, acetone	Filter paper	Passive adsorption/ thermal desorption	Elite-1 (30m × 0.25mm × 1.0µm)	He	MS	NIR	NR	[62]
34	Petrol, diesel, mineral spirits, paint thinner, paint remover, lighter fuel, turpentine	Carpets	SPME	Rtx-5MS (30m × 0.25mm × 0.25µm)	He (2ml/min)	MS	270°C	45-200	[63]
35	Petrol	Human hand	Passive adsorption/ elution	HP-5MS (30m × 0.25mm × 0.25µm)	He (1ml/min)	MS	250°C	10-450	[64]
36	Gasoline, diesel	NR	NR	HP-1 (25m × 0.20mm × 0.50µm)	He (1ml/min)	MS	280°C	NR	[65]
37	Petrol, kerosene	Carpet, wood	Passive adsorption/ elution	DB-5MS (30m × 0.25mm × 0.25µm)	He (1ml/min)	MS	250°C	50-550	[66]
38	Petrol, diesel, barbecue lighter, turpentine, industrial solvents	Soil, sawdust	HSSE, SPME	VF-23MS (30m × 0.25mm × 0.25µm)	He (1ml/min)	MS	NIR	40-400	[67]

39	Petrol, diesel	Human hand	Passive adsorption/ elution	AT-1 (30m × 0.25mm × 0.25µm)	He (3.2ml/min)	MS	NR	30-300	[68]
40	Petroleum distillates (gasoline, kerosene, charcoal lighter fluid, paint thinners)	Carpet, paper, wood, plastic, cloth	Dynamic adsorption/ elution	DB-5 (30m×0.25mm×1 µm) DB-225 (30m×0.25mm×0. .25µm)	H ₂ (1ml/min)	FID	280°C, 280°C	NA	[71]
41	Petrol, kerosene, diesel, white spirit, charcoal lighter fluid, paint thinners	Wood, carpet, plastic, clothes, painted surface	Dynamic adsorption/ elution	DB-5 (30m × 0.25mm × 1µm)	He (0.9ml/min)	MS	250°C	50-200	[73]
42	Petrol, diesel, kerosene	Soil, wood, paper	SPME	HP-5MS (30m × 0.25mm × 0.25µm)	He (1ml/min)	MS	280°C	35-650	[74]

ITMS in ion selective mode. They concluded that HS-SPME is very sensitive, simple and rapid technique for the extraction of small amounts of accelerants from fire debris [48].

Lentini and Armstrong compared the eluting efficiencies of carbon disulfide and diethyl ether and suggested that carbon disulfide is best solvent to elute compounds of interest from adsorption packages prior to analysis GC-FID but when GC-MS is used, diethyl ether is better choice of solvent than carbon disulfide because its eluting efficiency is comparable to carbon disulfide and it poses a much smaller health risk to fire debris laboratory personnel [49].

Gilbert described the potential use of extracted ion profiles in distinguishing an ignitable liquid from interference due to pyrolysis or other contaminants and concluded that individual extracted ion profiles are better than summed extracted ion profiles in identifying ignitable liquids [50].

Tan et al. identify and classify petroleum based accelerants using GC-MS and multivariate pattern recognition techniques such as Principal Component Analysis (PCA) and Soft Independent Model Classification Analogy (SIMCA). Detection limits of correct classification depends on sample class and were in the range of 5-50µl. They suggested that carpet is better source than wood for collection of accelerant residues. They concluded that PCA and SIMCA can be successfully applied to classify accelerants after GC-MS analysis [51].

Borusiewicz studied the effect of fire extinguishers such as foam, powder and snow extinguisher on detection and identification of diesel in arson debris and suggested that fire fighting action of fire extinguishers should be rapid as this

factor plays a significant role in recovery of accelerants from fire debris samples. They concluded that these fire extinguishers did not cause any interference in isolation, detection and identification of accelerants from fire debris samples [52].

Cavanagh et al. studied the background interferences generated from car carpets. They also investigated the persistency of petrol on carpet. They observed that deposition of target compounds onto carpet depends on occupation and behaviour of occupants and suggested that presence of larger volumes of petrol on carpet is an indicative of intentional addition of petrol to vehicle interior. They concluded that car carpets exhibit some of the compounds that may interfere with the detection and identification of petrol residues [53].

Zadora et al. identify petroleum products in arson debris and water by GC-MS and concluded that present method is rapid, sensitive and useful for analysis of samples even after several months of their collection [54].

Doble et al. classify premium and regular gasolines using PCA. They compared PCA and ANN's for classification of premium and regular gasolines from their GC-MS chromatograms. They concluded that PCA is a potential statistical tool to classify gasoline samples into either premium or regular class while ANN's are reliable and accurate statistical tool to classify gasolines into premium or regular groups and into seasonal formulation based subgroups [55].

Lloyd and Edmiston investigated the extraction efficiency of SPME fibers (PDMS and Carboxen/PDMS) used for extraction of hydrocarbons from fire debris samples and concluded that Carboxen/PDMS fibers are more efficient for the extraction of aromatic hydrocarbons compared to aliphatic hydrocarbons [56].

Sandercock and Du Pasquier identify and differentiate 35 gasoline samples using GC-MS after extracting trace polar and polycyclic aromatic hydrocarbon compounds using solid phase extraction. Linear discriminant analysis divides 35 samples into 32 unique groups. They concluded that polycyclic aromatic hydrocarbons can be used to distinguish between different gasoline samples due to their significant variation from sample to sample [57].

Almirall and Furton characterize the background and pyrolysis products that were generated during control burning of different substrates. They observed that many substrate backgrounds, combustion or pyrolysis products were also target compounds of ignitable liquid residue mixtures and could interfere

with identification process. They suggested that negative control samples of substrates present at fire scenes should be collected and analysed before actual samples of interest [58].

Barnes et al. compared different gasoline samples by GC-MS and sequential peak ratio method and target ion response and observed that wood does not cause any interference in comparison process. They concluded that such comparisons are useful in comparing gasoline (extracted from debris) to unevaporated gasoline samples and in associating evaporated gasoline sample with its source and discriminate from other sources of gasoline [59].

Cavanagh-Steer et al. investigate the transfer and persistency of petrol on car carpets. They observed that evaporation level of petrol increases with rise in time between transfer and analysis and concluded that presence of petrol (fresh or slightly evaporated) in a significant amount on motor vehicles carpet is an indicative of addition of petrol to vehicle interior and eliminates the potential contamination due to normal vehicle usage [60].

Borusiewicz et al. studied the effect of different factors (type of burned material and accelerant, time of burning, availability of air) on the detection of traces of accelerants. No significant relationship between time of burning and accelerants traces detectability was observed. They concluded that type of burned material causes highest interference in identification process while accelerant type and burning time is less significant. They also concluded that variation in air availability didn't significantly affect the detection of accelerants traces [61].

Borusiewicz and Zieba-Palus compared the adsorption efficiency of Tenax TA and Carbotrap 300 and concluded that Tenax TA is more effective for adsorption of non polar, high boiling compounds while Carbotrap 300 is more effective for adsorption of polar and volatile compounds [62].

Lu et al. compared the performance of differential mobility spectrometry (DMS) and mass spectrometry (MS) in the detection and classification of ignitable liquids from fire debris using projected difference resolution (PDR) and concluded that performance and efficiency of GC-MS is better than GC-DMS [63].

Montani et al. compared the efficiency of different matrices to collect the gasoline on suspects hands. They also developed the simple and effective sampling kit which was efficient in preventing external and cross contaminations. They concluded that non powdered latex gloves were most efficient and reliable in collecting residues of gasoline from suspect's hands and emit least interfering volatiles

[64].

Choodum and Daeid developed and validate a GC-MS method to analyse hydrocarbon residues encountered in fire debris samples. They also optimized the separation conditions such as carrier gas flow rate, column temperature programming and inlet temperature and concluded that present method is rapid, sensitive and provides better resolution than recommended National Centre of Forensic Science (NCFS) method for ignitable liquid analysis [65].

Prather et al. compared the simulated ignitable liquid residues to corresponding liquid in the presence of both evaporative losses and matrix interferences using multivariate statistical procedures such as Pearson Product Moment Correlation Coefficient (PPMC), Hierarchical Cluster Analysis (HCA) and Principal Component Analysis (PCA) and concluded that these statistical procedures can be used to associate ignitable liquid residues to liquid standards even in the presence of evaporation and matrix interferences but not to a specific level of evaporation [66].

Cacho et al. developed a novel method for the separation and identification of combustion accelerants in fire debris by GC-MS. Simple headspace sorptive extraction (HSSE) technique was used to preconcentrate the analytes. They also compared HSSE procedure with SPME procedure. 1g of debris was placed in 15ml glass vials for HSSE procedure. PDMS stir bar was exposed to vial headspace for 60 mins at 50°C. Commercial stir bars coated with 0.5mm thick layer of polydimethylsiloxane were used as extracting phase. Thermal desorption unit (TDU) equipped with an autosampler and a programmed temperature vaporization (PTV) cooled injector system was used for sample introduction into GC. Limit of detection was in the range of 0.1-0.7ng/g depending on nature of particular accelerant. They concluded that HSSE procedure is more efficient and sensitive method than SPME and can be applied to extraction of accelerants from fire debris [67].

Muller et al. developed a simple, sensitive and innovative method for the identification of trace amounts of ignitable liquid residues on hands of suspect even after 3h of moistening. Method cannot distinguish between fuel used as an arson accelerant and fuel used for legitimate use [68].

Smale et al. compared the sensitivity and efficacy of different techniques used to extract ignitable liquid residue from concrete. Three of these techniques involve the covering of concrete surface with an absorbent material such as cat litter, absorbent

matting and cotton pads while fourth technique uses Passive Headspace Residue Extraction Device (PHRED). In these techniques, absorbent material was applied to concrete and allowed to absorb any ignitable liquid residue for 1hr. The absorbed components were extracted using passive headspace extraction. PHRED was affixed directly to concrete surface and then evaporates ignitable liquid residue directly from concrete to charcoal strip within the device by heating. They concluded that PHRED is more sensitive and effective technique than cat litter in extracting ignitable liquid residue from concrete and it is portable [69].

Waddell et al. generates a clusters based on chemical composition of ignitable liquids by applying hierarchical cluster analysis on total ion spectrum data and classify them into 2 major groups: aliphatic and aromatic [70].

Multidimensional GC Methods

Jayatilaka and Poole [71] and Taylor et al [72]. identify petroleum distillates in simulated arson samples by multidimensional gas chromatographic method and concluded that present method is less influence by interfering co-eluting and matrix components and can be used to identify petroleum distillates in contaminated arson samples with improve certainty.

Pert et al. also advocate the utility of two dimensional GC (GC×GC) in the analysis of fire debris because it eliminates the interferences due to pyrolyzed products [10].

Conclusion

Test and control samples should be collected from crime scene and must be store in recommended containers in order to get valuable information from them. Both conventional and modern extraction procedures have their own merits and demerits and are used on the basis of availability. It is concluded that no single extraction method is universally effective for isolation of different petroleum products from variety of substrates of forensic importance.

Different spectroscopic methods used to analyse fire debris residues cannot provide the pin point identity of petroleum products due to their non specific nature.

However, these methods can be successfully used for screening purpose at initial stage of analysis.

Advances in instrumentation open the doors for utilization of more sophisticated analytical techniques in the analysis of fire debris residues. GC-MS is a well established technique for the analysis of fire debris residues due to its sensitivity, selectivity and reproducibility.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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