MSI Journal of
Multidisciplinary Research (MSIJMR)

A Systematic Review On the Occurrence, History, Effects, Analysis of POPs And its Future Aspects

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The authors declare that no funding was received for this work.

Received: 09-February-2026
Accepted: 05-March-2026
Published: 09-March-2026

This article is published in the **MSI Journal of Multidisciplinary Research (MSIJMR) ISSN 3049-0669 (Online)**

The journal is managed and published by MSI Publishers.

Volume: 3, Issue: 3 (March-2026)

ABSTRACT: Persistent Organic Pollutants (POPs) are toxic substances and may not be biodegraded in the environment and accumulate in the organisms. Environmental forensic investigations are aimed at identifying and studying these pollutants in different matrices in the forms of biological samples, air, water, and soil. This paper will focus on identifying the important processes in sample preparation in order to identify POPs in environmental forensic analyses. The sample preparation procedure is divided into a number of stages: sampling, sample extraction and purification. Some of the common methods of extraction are Solid Phase Extraction (SPE), Microwave Assisted Extraction (MAE) and Accelerated Solvent Extraction (ASE). Recent technologies like the QuEChERS and passive sampling devices have also been implemented in order to enhance the efficiency and accuracy of the process. Proper sampling can guarantee good recovery; less solvent was used and the integrity of the analytes. Such procedures are important to present the accurate and dependable findings in the legal and regulatory settings, and make the forensic information defensible. Preparation of

samples continues to be an integral part of environmental forensic research, which will increase the accuracy and dependability of data in the detection of POPs. The further improvements in the extraction methods lead to the more efficient and environmentally friendly forensic science.

Keywords: *Persistent Organic Pollutants (POPs); Environmental Forensics; Sample Preparation; Solid Phase Extraction (SPE); Microwave Assisted Extraction (MAE); Accelerated Solvent Extraction (ASE).*

Graphical Abstract:

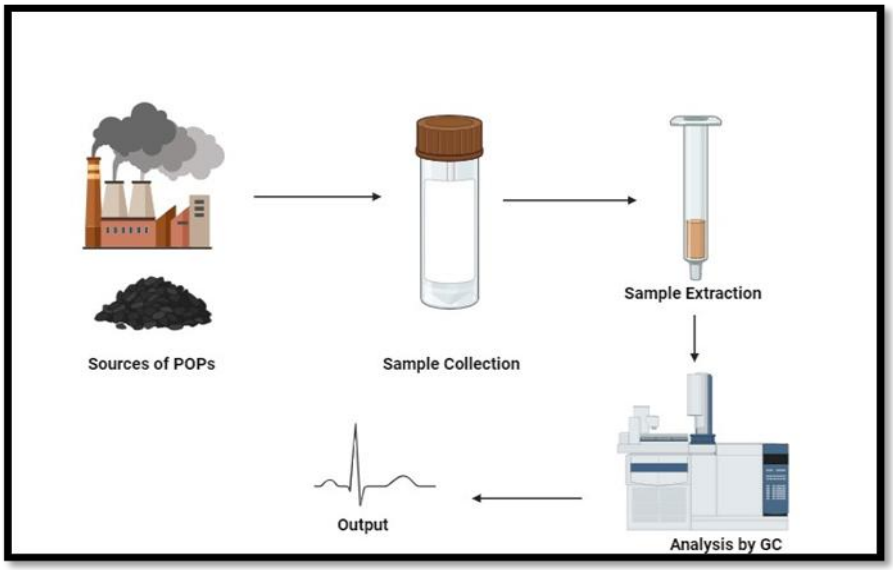

Figure 1 Graphical abstract

1 Introduction

Because forensic science uses a wide range of analytes and sample types to enhance criminal investigations, it can be regarded as one of the widest analytical fields. Examinable material, for instance, may vary from intricate biological molecules like DNA to simple visual patterns like fingermarks in the realm of human identification alone [1] .One of the government's top priorities right now is environmental protection [2]. Urban environments are subjected to a multitude of detrimental natural and human-induced phenomena. The primary issues with conducting a forensic investigation of the ecological condition of the urban environment are examined in this scientific essay [3]. Urban development reformation led to a reduction in the scope of environmental competence, which became a negative

matter [4]. The goal of environmental forensics is to investigate pollution occurrences to determine the source of the pollution and any effects it may have had on the environment or public health [5] . In order to confirm the presence of contaminants, casework heavily depends on field investigations and subsequent laboratory-based analysis [6] .All ecosystems on Earth contain toxic compounds that have an impact on water sources, agricultural productivity, and biodiversity. Furthermore, scientists believe that a significant number of chemical pollutants with unknown health effects are carried by every human body [7]. POPs are among the many compounds that may move great distances by air, water currents, and migrating species. These chemicals have been discovered in places where they are not utilized, and more significantly, they can stay in the human body for over 50 years. Moreover, there are issues with their sources and releases, which differ greatly [8] Figure S2 shows the Major sources of persistent organic [9]. Persistent organic pollutants, or POPs, have been widely produced since the 1950s. These POPs have gotten into the environment through manufacture, usage, and disposal. POPs that are poisonous, bacchanals ante fatty tissues of humans and animals, and are difficult to break down are referred to as the "dirty dozen." In accordance with the Stockholm Convention, the United Nations Environmental Programmed (UNEP) has officially registered these pollutants [10].

The cycling of persistent organic pollutants (POPs) in the Earth's surface system depends on the atmosphere, which also offers vital information regarding the origins, distribution, and transmission of POPs. Highly volatile POPs (HV-POPs) are one new POP that has not received enough attention in POP research. The volatilities of POPs range from ELVOCs to VOCs, but atmospheric chemistry lacks more precise operational boundaries [11]. This ignorance contributes to a lack of knowledge about the destiny and health concerns of persistent organic pollutants (POPs) and causes a disconnection between POPs and secondary organic aerosols (SOA), which may be caused by hydrophilic oxidized POP compounds [12]. Persistent organic pollutants (POPs) were shown to be substantially linked to both the risk and survival of amyotrophic lateral sclerosis (ALS) in a Michigan research [13]. There were 164 ALS sufferers and 105 control individuals in the research. Eight out of twenty-two polychlorinated biphenyls and seven out of ten organochlorine pesticides (OCPs)

were shown to be substantially linked to ALS. Alpha-hexachlorocyclohexane, hexachlorobenzene, trans-noncolor, and cis-noncolor were among the OCPs that collectively caused a 2.58-fold increase in the risk of ALS. Additionally, the total mixing of all POPs increased the incidence of ALS death by 1.65 times [14].

POPs basically have four things in common: For POP separation in GC analysis, capillary columns with non-polar or slightly polar stationary phases are usually utilized. For the detection of halogenated chemicals, detectors with high sensitivity and specificity include mass spectrometry (GC-MS) and the electron capture detector (ECD). While quantitative analysis entails the development of calibration curves for precise measurement, qualitative analysis compares retention durations and mass spectra with recognized standards. Replications, blanks, and standards are only a few of the quality control techniques that guarantee the correctness and dependability of the findings [15]. Compounds with specific physical and chemical properties that allow them to persist for extended periods of time are known as persistent organic pollutants (POPs). Examples of these compounds include polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), polychlorinated dibenzodioxins/dibenzofurans (PCDD/Fs), PBDDs, polybrominated diphenyl ethers (PBDEs), hexachlorocyclohexane isomers (HCHs), and others [16]. POPs bioaccumulate due to their persistence, which could have a major negative influence on the environment and human health. POPs can accumulate in living organisms due to their lipophilicity; this effect is particularly pronounced in animals that occupy higher positions in the food chain. Higher levels of POP preservation can be found in foods heavy in fat, like milk, eggs, fish, and meat. Humans may be exposed to POPs from the contaminated environment in addition to exposure from the foods they regularly eat [17].

POPs are primarily found in soils and sediments where they partition into organic materials, accounting for most of their environmental load. Concentrations in "adjacent" media, such as air or water, would be significantly impacted by small changes in the bulk of soils or sediments. POP concentrations and loads in soils and sediments are influenced by a variety of processes, such as biodegradation, volatilization, and "ageing" impacts, which over time may result in less or non-

extractable leftovers. POPs built up in aquatic animals, causing them to perish [18] .Exposure to these substances may be linked to birth abnormalities, immune system and reproductive system problems, some malignancies, and even decreased intelligence [19]. The Stockholm Convention, which was ratified in 2001 and went into effect in 2004, requires state parties to take actions targeted at lowering and eventually doing away with the discharge of POPs into the environment in response to the worldwide threat and health hazards posed by POPs. [20]The requirement for forensic laboratories to meet complete testing demands has led to challenges related to the growing range of analytical techniques and instruments. These have driven the quest for novel and flexible procedures that can detect, identify, and quantify analytes of forensic interest [21]. POPs can exhibit a variety of physio-chemical properties as well as matrix interferences, making their analysis in environmental matrices complex. As a result, highly selective, sensitive, accurate, and precise detection techniques are needed, frequently in conjunction with time-consuming cleanup procedures, especially when it comes to the analysis of PCDD/Fs, PCBs, and PBDEs in sediments [22].

For many different types of samples including organic analytes, gas chromatography coupled with mass spectrometry (GC-MS) delivers analytical information that is conclusive. Field portable GC-MS systems have been crucial to important environmental and forensic applications within the past ten years. Since the middle of the 1970s, gas chromatography (GC) in conjunction with high resolution mass spectrometry (HRMS) has been the "golden standard" for this analysis [23]Two-dimensional (2D) gas chromatography (GC × GC), a multidimensional approach, has a superior separation space that allows component peaks to spread out across new coordinates, lowering the chance of overlap, making it a better fit for this type of study. During sample elucidation, a significant benefit of 2D methods over conventional 1D procedures can be seen. Comprehensive 2D approaches enable the transit of the full sample through the system, resulting in adequate identification and quantification of the analyte, while standard GC techniques only display data on a portion of the sample [24].

1.1 Sources of POPs

A class of hazardous substances known as persistent organic pollutants (POPs) linger in the environment, bioaccumulate in living things, and endanger both ecosystems and human health. Effective pollution control and mitigation initiatives depend on locating POP sources. With an emphasis on industrial chemicals, this article explores the many sources of POPs and offers in-depth analysis based on current research. Organic pollutants are mostly produced by industrial activity [25]. The term "polychlorinated biphenyls" (PCBs) refers to a class of synthetic organic compounds made up of 209 distinct congeners, or individual chlorinated compounds. PCBs were widely employed because of their non-flammability, chemical stability, and insulating qualities in a variety of industrial applications, such as coolants and insulating fluids in transformers and capacitors. On the other hand, PCBs are extremely persistent in the environment and have been connected to a host of health issues, including as neurological damage, cancer, and immune system suppression. Due to their long-term stability and bio accumulative nature, PCBs are still found in the environment even though their manufacturing and usage were outlawed or severely restricted in many countries in the late 1970s and early 1980s [26]. Figure S3 shows the sources of POPs [27]. A class of chemical molecules made up of many aromatic rings is known as polycyclic aromatic hydrocarbons, or PAHs. For example, a study on urban air pollution discovered that vehicle emissions were primarily responsible for nearly 60% of the overall amounts of PAHs, with diesel engines accounting for a large portion of these emissions [28]. The petrochemical sector is one industrial source of volatile organic compounds (VOCs), as the refining and burning of petroleum products produces significant amounts of benzene and toluene (Journal of Environmental Science, 2022. Pesticides used to manage pests, such as DDT, atrazine, and chlorpyrifos, can linger in the environment and have an impact on species that are not intended targets [29].

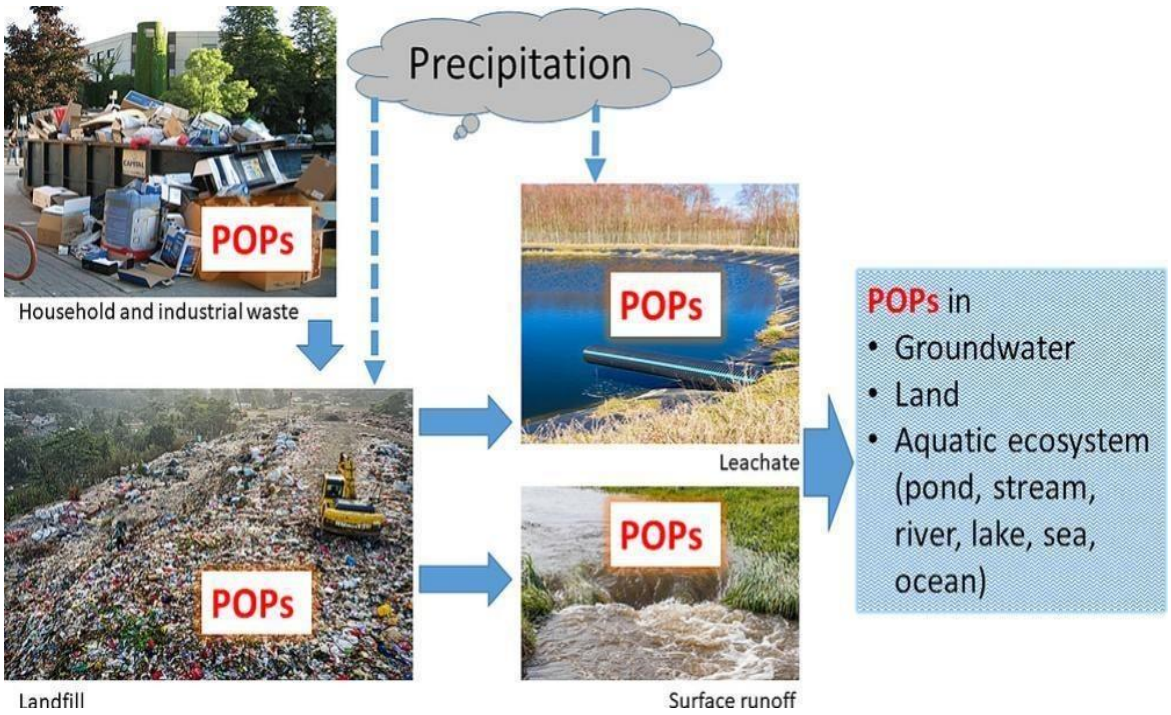

Figure 2 Major sources of persistent organic pollutants [9].

Table 1 Persistence Organic Sources are Detailed Discussed with Description, Examples, Quantity Data, Quality Data and Analysis

Sources	Description	Examples	Quantity Data	Quality Data	Analysis	References
Industrial Processes	Release POPs into the environment through manufacturing and waste incineration.	PCBs, dioxins, chlorinated solvents	Millions of tons of POPs released annually.	Regulatory data indicates frequent exceedance of permissible limits for POPs.	Industrial activities contribute to millions of tons of POPs annually, impacting ecosystems and human health.	[30]
Agricultural Activities	Major source of POPs due to pesticide and herbicide use, with residues leaching into water	DDT, atrazine, glyphosate	Significant percentage of global POPs emissions.	Monitoring data shows contamination levels often exceed safe thresholds for POPs.	Agricultural runoff contributes to a significant percentage of global POPs emissions,	[31]

Sources	Description	Examples	Quantity Data	Quality Data	Analysis	References
	bodies.				posing risks to aquatic ecosystems.	
Urban Runoff	Carries pollutants like PAHs and heavy metals into water bodies, impacting aquatic life.	PAHs, heavy metals, phthalates	Substantial loads of POPs in receiving waters annually.	Water quality assessments often exceed recommended standards for POPs.	Urban runoff contributes substantial loads of POPs to receiving waters, threatening water quality and biodiversity.	[32]
Combustion Processes	Releases POPs like PCDDs and PCDFs into the atmosphere, affecting air quality and human health.	Polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs)	Significant quantities of POPs emitted annually.	Air quality monitoring often shows exceedance of standards for POPs.	Combustion sources emit significant quantities of POPs annually, leading to health risks for nearby populations.	[33]
Industrial Chemicals	Chemical spills and improper disposal lead to POPs contamination of soil and groundwater.	Perfluorinated compounds (PFCs), polybrominated diphenyl ethers (PBDEs)	Major contributor to global POPs emissions.	Soil and groundwater sampling data often exceed regulatory limits, indicating poor waste management.	Industrial chemicals contribute to the majority of POPs emissions globally, necessitating stringent waste management practices.	[34]

Sources	Description	Examples	Quantity Data	Quality Data	Analysis	References
Pesticides and Herbicides	Runoff and leaching of pesticides contribute to POPs pollution in surface water.	Organophosphates, carbamates, chlorpyrifos	Substantial amounts of POPs released into the environment.	Ecotoxicological studies show adverse effects on aquatic organisms.	Pesticide use results in the release of substantial amounts of POPs into the environment, posing risks to aquatic ecosystems and human health.	[35]
Household Products	Disposal of consumer products releases POPs into soil and water, impacting ecosystems.	Triclosan, phthalates, alkylphenols	Significant volume of household products containing POPs.	Effluent monitoring data often reveals contamination in aquatic ecosystems.	Household products contribute to POPs contamination, necessitating proper disposal and eco-friendly alternatives.	[36]
Pharmaceuticals and PPCPs	Disposal of pharmaceuticals contributes to POPs contamination of surface water.	Antibiotics, hormones, sunscreen chemicals	Widespread use and disposal contributing to POPs pollution.	Toxicological studies indicate potential health risks, highlighting the need for regulation.	Pharmaceutical and PPCP pollution poses risks to aquatic organisms and human health, emphasizing the need for proper disposal and regulation.	[37]

2 Material and Methods

2.1 Sample Preparation

2.1.1 Collection of samples

An effective sampling procedure is essential to any inquiry involving environmental forensics. If data are to be defended in court, a number of factors that could skew the data and provide incorrect conclusions must be understood [38]. Understanding the compound's environmental chemistry is crucial for any inquiry so that the right matrix can be targeted. In light of the question addressed, consideration should also be made to creating a suitable sample strategy. Some frequently asked questions are: Was the animals impacted by the contamination, was a regulatory threshold limit broken, and what are the possible sources and levels of contamination? The answers to each of these queries can require gathering various samples for a particular objective [39]. During the course of an environmental forensics examination, many samples are collected. These could be gathered from a wide range of diverse matrices, including flora, soils, water, animals, air particles, and gas phases. Samples must always be properly labeled and kept in containers that are suitable for the type of analysis being done. Every sample needs to be sent to the lab as soon as possible, ideally in less than 24 hours, and with the proper chain of custody paperwork. Inadequate adherence to the relevant best practice guidelines may result in data inaccuracies and, in the end, the dismissal of the findings from the court [40].

2.1.2 Extraction of sample

To extract the chemicals of interest from the bulk matrix, extraction of the sample is an essential step. Internal standards are usually introduced to the sample before extraction to quantify the recovery rate and extraction efficiency as well as to enable quantification and quality control (QC). Because they react similarly to the native chemicals in the sample, isotopically labelled analogues of the target compounds are preferable because they provide the most exact and accurate data for calibration and quantification. Isotope-labeled solutions, however, can be highly costly, and there may not always be considered standards are available[41].

Recovery rates can be computed in these situations using a representative compound that shares characteristics with the target chemical[42].

2.2 Air, aqueous and liquid matrices

The traditional method of extracting liquid/liquid samples, such as biological fluids, is through aqueous and other liquid samples. If samples contain particles, they can be filtered to separate the precipitates/particles; depending on the project's goals, the two fractions can then be determined independently or combined again later. This is especially crucial for hydrophobic substances like BDE 209, which are mostly found in the particulate phase. Solid phase extraction is an increasingly popular substitute for liquid/liquid extraction (SPE). This method separates non-polar analytes, such as dioxins, from polar liquids using a stationary phase or resin in an extraction cartridge or disc. Particles can also become stuck on top of the extraction bed or disc when samples are extracted using SPE [43]. Particle and disk quantitative elution can be carried out in many steps to separate different classes of compounds, or in a single step for a large number of analytes, which can drastically cut down on solvent consumption and analytical time [41]. Other techniques that have been effectively employed for POP extraction include solid phase micro extraction (SPME) , hollow fiber liquid-phase microextraction (HF-LPME) , stir-bar sorptive extraction (SBSE) with thermal desorption directly into the gas chromatograph [44], and passive samplers like polyethylene strips or semipermeable membrane devices (SPMD) [45]. In more recent times, scientists have also sampled the aquatic and atmospheric environments using passive samplers, such as low-density polyethylene passive samplers (LDPE-PAS). The advantages of PAS include low to no infrastructure requirements, simple extraction, extended deployment timeframes, and large POP capacities [45].

2.3 Solid Matrices

Soxhlet extraction or Soxtec, an automated Soxhlet variant, have traditionally been used to extract solid matrices (such as soil/sediment, biota, or vegetation). There have also been uses for sonication, microwave aided extraction (MAE) [46], and pressurized liquid extraction (PLE), also referred to as accelerated solvent extraction

(ASE) . Compared to Soxhlet and sonication, PLE and MAE are automated methods that expose the samples to high temperatures and pressures, which can lead to faster and more effective extractions. Vazquez-Roig and Picó have recently examined the application of PLE for the extraction of POPs in environmental samples [47]. Using supercritical carbon dioxide, supercritical fluid extraction (SFE) is a process that requires very little solvent to elute the analytes from the carbon or C18 trapping material and no solvent for extraction [48]. However, caution must be exercised when using an acid or base as they may breakdown potential compounds of interest such as BEHTBP and other sensitive HFRs. Solid matrices can also be digested using an acid or base and subsequently extracted using SPE or liquid/liquid extraction. Many of the methods covered in section 2.2 are somewhat costly because they take a long time and necessitate the use of an experienced scientist in order to yield satisfactory results [49],[50]. A relatively clean injection-ready extract is produced by extracting the sample in a disposable tube with a dispersant present, though extra cleanup procedures are frequently required. The approach, which was first created to detect veterinary pharmaceuticals, has been adapted to detect non-polar analytes such as PCBs and PAHs. It is a great option for preliminary sample screening [51],[52]. A review has been conducted on the application of QuEChERS in identifying persistent organic contaminants in environmental matrices [53],[54]. Novel materials known as metal-organic frameworks (MOFs) can be functionalized to provide desired host-guest interactions. Figure S4 shows the schematic illustration of Metal organic frameworks as an adsorbent [55].

Table 2 Summary of extraction techniques

Method	**Main Matrix**	**Brief Overview**	**References**
Solid Phase Extraction (SPE)	Liquid/Aqueous	Analytes are extracted from liquids by passing a sample through a stationary phase or resin in an extraction cartridge or disc. After that, a solvent can be used to gather these.	[56]
Stir-bar Sorptive Extraction (SBSE)	Liquid/Aqueous	To extract analytes from the liquid mixture, a tiny bar coated with sorbent	[57]

Method	Main Matrix	Brief Overview	References
		is added and agitated. After that, a solvent can be used to gather these.	
Hollow Fiber Liquid Phase Microextraction (HF-LPME)	Liquid/Aqueous	Sample is retained after being passed through a tiny hollow fiber that has been treated with adsorbent. After that, a solvent can be used to gather these.	[58]
Solid Phase Micro Extraction (SPME)	Liquid/Aqueous	To extract analytes, a fiber coated with an adsorbent is placed into the sample. After that, either the fiber can be put straight into the injector, or they can be gathered using a solvent.	[59]
Semipermeable Membrane Devices (SPMD)	Liquid/Aqueous	Usually, the sampler is submerged in a watercourse and allowed to absorb analytes for a while. A solvent can then be used to remove these from the membrane.	[60]
Microwave Assisted Extraction (MAE)	Solid	Samples are put in a temperature-controlled container, and analytes are extracted into a solvent using microwave digestion.	[61]
Supercritical Fluid Extraction (SFE)	Solid	After supercritical CO2 is pressurized to dissolve samples in a temperature-controlled vessel, the gas is released, causing the analytes to precipitate in a collector containing a small amount of solvent.	[62]
Accelerated Solvent Extraction (ASE)	Solid	To extract analytes, a cartridge-containing sample is filled with solvent and forced through at a specific temperature and pressure.	[63]

2.4 Sample Analysis:

For precise measurement, chemicals need to be completely separated from one another. In a quantity of the cases, selective detection might make this feasible. Although a number of chromatographic methods are High performance liquid chromatography (HPLC) and gas chromatography (GC) are potentially helpful techniques. Beginning in the year 2000, Many environmental separations have also been successfully accomplished with ultra-high performance liquid chromatography (UHPLC), mostly as a means of boosting laboratory throughput [41].

Table 3 A synopsis of the Table presents distinct strategies for separation

Technique	**Merits**	**Demerits**	**Analytes Examined by Use of the Technique**	**References**
High Performance Liquid Chromatography (HPLC)	A large variety of possible analytes, including polar, ionic, non-volatile, and thermally labile analytes. • Brief run durations • The complementarity and diversity of possible ionization methods.	Less theoretical surfaces mean a lower potential for separation than GC. Compounds that are too non-polar may bond too tightly to the column, resulting in slow elution and ineffective separation.	OC pesticides, PBDEs, HBCD, Perfluorinated chemicals.	[64, 65]
Gas Chromatography (GC)	Outstanding separation potential	Limited to use with more flammable substances.	OC pesticides, PCBs or PCNs, PCDD/F, PBDEs, HBCD, Perfluorinated chemicals.	[66] [67]
Multidimensional Chromatography	• Good separation potential	Limited to usage with more volatile substances. Frequently requires larger files and further data processing.	OC pesticides, PCBs or PCNs, PCDD/F, PBDEs	[68, 69]

2.5 Gas Chromatography

The main reason why chemicals in a mixture separate when utilizing gas chromatography is because of each compound has seven distinct boiling points, and each one interacts differently with the column's stationary phase. Recognizing the impact of stationary phase chemistry Predicting GC separation is not that difficult, and commercial software packages like "Pro ez-GC" can be used to conduct calculations. These applications can be quite beneficial for both column selection and separation optimization [41]. Figure S5 shows the simple Instrumentation of Gas Chromatography [70].By maximizing the chemicals' maximum potential resolution inside the chromatographic space, which reduces the strain on detecting systems and raises the standard of the analysis [50].

2.5.1 Column selection

The capillary column is the GC's core. The features of separation are determined by the choice of stationary phase, column size, and carrier gas (velocity). Because most POP combinations have complicated compositions, most research have attempted to maximize the chromatography's resolution to separate the greatest number of pollutants. Most carrier gasses use hydrogen and helium, and hydrogen offers the best resolution at the fastest carrier gas velocities. The stationary phases utilized in halogenated pollutant GC are displayed in Table 2. Non-polar to slightly polar stationary phases, including DB-1, DB-5, BPX-5, HT-8, CP-Sil8CB-MS, or CP-Sil-19, are the most commonly employed [71, 72]. The normal film thickness is 0.25 micrometers. 30- to 60-meter columns are typical, yet there are situations where shorter columns are advantageous. The resolution is directly correlated with the column diameter. Conventional column diameters fall between 0.25 and 0.32 mm, yet for the same column length, narrow bore columns (0.10-0.15 mm) offer significantly more theoretical plates [73, 74].

Table 4 Stationary phases used for POPs Analysis

Polarity Scale	Stationary Phases	Reference
5	100% Dimethyl polysiloxane	[75]
8	5% Phenyl-(arylene)–95% methyl polysiloxane	[76]

Polarity Scale	Stationary Phases	Reference
17	50% Phenyl–50% methyl polysiloxane	[77]
24	75% Phenyl–25% methyl polysiloxane	[78]
43	50% 3-Cyanopropyl–50% phenylmethyl polysiloxane	[79]
52	Polyethylene glycol	[80]
88	100% 3-Cyanopropylpolysiloxane	[81]
Non polar	50% n-Octyl–50% dimethyl siloxane	[82]
Moderately polar	65% Phenyl–35% methyl polysiloxane	[76]
	Cross-linked methyl–phenyl–polysiloxane block polymers	
Polar	Polysilphenylene phase	[83]
	44% Methyl–28% phenyl–20% cyanopropyl polysiloxane	[84]
	Biphenylcarboxylate ester methylpolysiloxane	[85]
	Dimethyl (50% liquid crystal) polysiloxane	[86]

2.5.2 Multidimensional Chromatography

At the close of the 20th century, comprehensive two-dimensional gas chromatography (GCxGC) was established by the late John Philips, in the third century. The method is superior to single-dimensional chromatography in a number of ways [87].

2.5.3 Instrumentation

Two GC columns of GCxGC attached with modulator, a thermally or valve-controlled device that connects nine distinct phases, captures compounds from the first column, generally of a complimentary phase and re-injects them in a tight band onto the second column where additional separation takes place [88, 89]. The ability to record a small number of measurements is one of this technique's biggest hurdles over the roughly 400 ms wide, extremely tiny peaks created during the modulation process. About 20 spectra per second are needed to precisely characterize a second dimension GC peak [41]. Figure S6 shows GC x GC-ECD chromatogram for POPs determination of a sediment sample [41].

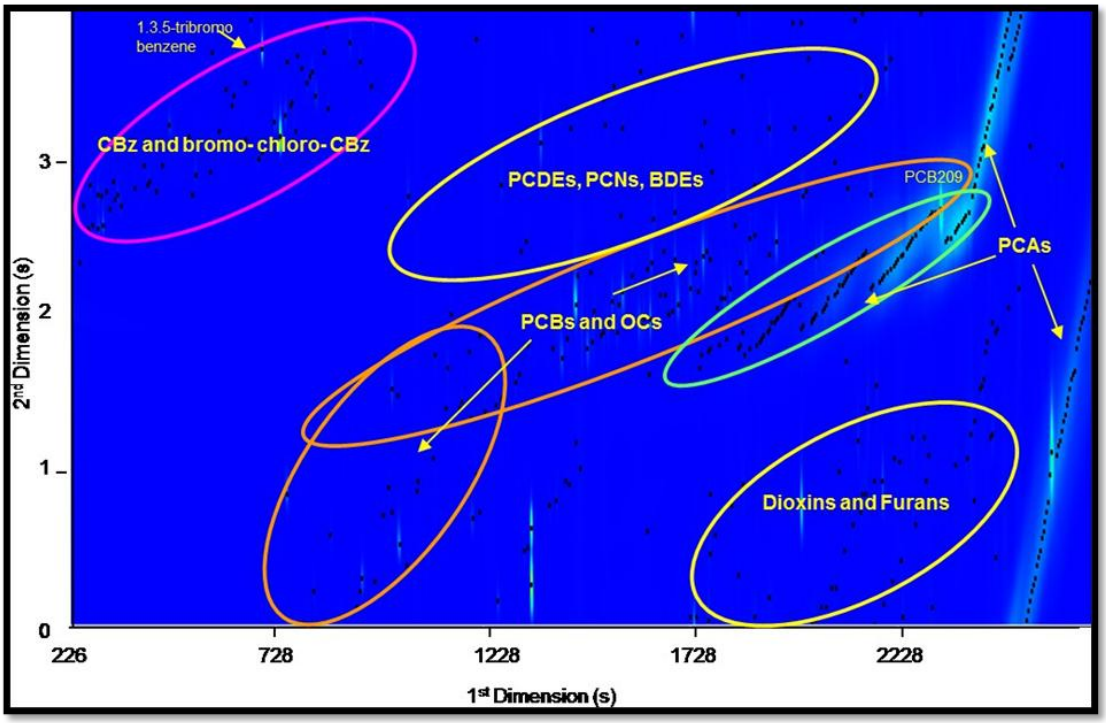

Figure 3 GCxGC-ECD chromatogram for POPs determination of a sediment sample [41].

2.5.4 Advantages of GCxGC

It can do eradicate with the requirement for multicolumn analysis or fractionation, and the modulation process yields a lot of chromatographic peaks that are taller and narrower, increasing signal-to-noise ratios (sensitivity) by as much as times larger than that. In addition to being employed for focused analysis GCxGC was employed in the non-target compound screening [90, 91]. The intricate designs found in the GCxGC chromatograms may also be utilized to identify contamination sources by acting as fingerprints. Because GCxGC generates congener-specific data, it is highly suitable for environmental forensics investigations including intricate biological matrices [50, 92].

2.6 Analyte Detection

There are numerous choices for detection and quantification after the chemicals of interest have been separated. When choosing a detector, the primary factors to consider are sensitivity, selectivity, and analysis speed [93, 94]. Mass spectrometry has recently made significant strides, leading to the development of commercially available instruments that are automated, highly selective, easy to use, and low maintenance and cost [95, 96]. As a result, for both screening and quantitative

applications, bench-top quadrupole and time-of-flight mass spectrometers have occasionally replaced FID and ECD. Most analytical laboratories now contain some type of high-resolution mass spectrometer (MS) or tandem mass spectrometer (MS) due to their increased accessibility. The ability to employ mass labelled internal standards, which can greatly improve accuracy and precision, is a primary benefit of mass spectrometers, a flexible detector with good sensitivity and dynamic range [41]. No other method can offer the same abundance of structural details of a chemical compound's few pictograms for qualitative work. N2 was originally used as a GC-ECD carrier gas in 1997 to separate different compounds that were dissolved in benzene. The goal of the study was to advance ECD technology by substituting a pulse-modulated approach for direct current ECD. This work reexamines the usage of 52 POPs from five classes of organic contaminants including chlorine as a replacement mobile phase for He. Organochlorine insecticides, chlordane, technical chlordane, toxaphene, and specific polychlorinated biphenyl (PCB) aromatic hydrocarbons were among the POPs. The objective is to verify and suggest a N2-based GC-ECD technique that can perform as well as or better than the He-based approach [75].

2.7 Ionization Techniques used in Mass Spectrometry

Ions can be produced, and mass analyses carried out using a variety of methods. The most widely used method for identifying POPs is electron ionization (EI), usually in conjunction with gas chromatography (GC). To compare spectra and identify non-target compounds, EI mass spectral libraries have been established. Under EI conditions, significant fragmentation happens, which is essential for the effectiveness of library matching. [97].

Because electron capture negative chemical ionization (ENCI) is selective for chemicals with a high electron affinity, it is especially useful for determining halogenated POPs [97, 98]. High electrical fields are used in close proximity to emitter needles to achieve field ionization. Molecules undergo photoionization when one or more ultraviolet (UV) or vacuum ultraviolet (VUV) photons are absorbed. Although these ionization methods greatly increase the sensitivity and selectivity of

detectors for aromatic compounds, laser-based photo-ionization technologies remain too costly and complex for [99].

2.7.1 Nominal mass resolution mass spectrometers

There are two general categories of mass analyzers: nominal mass resolution and high mass resolution. A mass spectrometer's resolution.

Equation 1

$$\mathbf{R} = \frac{M}{\Delta M}$$ [100].

Factors affect Nominal Mass Resolution

An ion guide collision cell is positioned between two quadrupole mass analyzers in tandem equipment. A quadrupole ion trap, time-of-flight, FT-ICR, or Orbitrap is used as the final mass analyzer in other hybrid kinds. The initial selection and focusing are done using the first quadrupole. Helium, nitrogen, oxygen, or argon are the colliding gases that are poured into the collision cell to create distinct pieces that are then mass separated by the final mass analyzer. Tandem mass spectrometry offers the benefit of being able to significantly minimise chemical noise, which allows it to filter out non-target ions and achieve low femtogram detection levels [101].

The majority of MS/MS instruments have a good linear dynamic range (4-6 orders of magnitude), which makes this method a great option for determining specific trace levels of pollutants in biota and food webs, like PBDEs and other POPs, The inability to detect non-target molecules and the complexity of developing and optimising MS/MS studies compared to single stage MS experiments are the drawbacks of MS/MS instruments [102, 103].

2.7.2 High resolution mass spectrometers

Although the magnetic deflection devices can perform tandem MS and HRMS, focused compound analysis in environmental and environmental forensics applications typically uses just the former mode. When combined with cryogenic zone compression gas chromatography, modern magnetic sector instruments can achieve low-femtogram instrument detection limits (IDLs) or even attogram IDLs.

These instruments are regularly calibrated to produce a resolution of >10,000 (10% valley) [104]. The TOF instruments have an easy-to-maintain, adjust, calibrate, and operate design. They come in three different resolutions: low resolution (7,000 to 10,000), mid resolution (7,000 to 10,000), and high resolution (> 20,000). The short dynamic range, often restricted to 4 orders of magnitude, is the main drawback of the majority of TOF devices. The TOF's main benefit is its high data acquisition rate (20–200 scans/second) for full-scan mass spectra recording. Because of this, they are the preferred detector for environmental forensics and quick GC [103, 105]. The length of the transient, or the amount of time the ions are confined during detection, determines the acquisition rate and resolving power of these sensors. Higher resolution is achieved with longer transients [72, 106].

Table 5 Comparison of different detectors used for the determination of POPs

Type of Detectors	**Mass Resolution**	**Cost of Analysis**	**Selectivity**	**Sensitivity**	**Polybrominated Diphenyl Ethers (PBDEs) and Hexabromobiphenyl**	**Hexabromocyclododecane (HBCD)**	**Reference**
ECD	na	low	Very low	high (halogenated) low (non-halogenated)	8-33 pg/g (fish)	6 pg/g (milk)	[107, 108]
qMS	Nominal	low	Low	Moderate	0.005-0.1 µg/L (water)	0.03 µg/L (water)	[109]
TOFMS	Nominal >20000	Low to high	High	Moderate to high	0.025-5 ng/g (fish)	0.9-4.5 pg/g (fish)	[22]
HRMS	>10000	High	High	High	<200 ng/g (soil)	5 ng/g (dust)	[110]
MS/MS	Nominal >20000	High	High	High	1-80 ng/g (dust)	1 ng/g (dust)	[111, 112]

3 Discussion

3.1 Developing, validating, and QA/QC methods

The scientific method needs to be testable and verifiable, published in a peer-reviewed journal or other comparable publication, have a defined rate or margin of error, be applied with the proper standards and controls, and have received substantial acceptance from the scientific community [73]. The most crucial prerequisite for any method is that it be suitable for the intended use, meaning that it meets all the requirements for data quality that the client needs in order to use the method's output to make informed judgments [113].

3.2 PCBs to act as an illustration for ecological criminology examinations

PCBs are one of the most generally concentrated on gathering of POPs; but there is no ordinarily involved strategy for 3 PCB assurance. PCBs were first recognized as obstructions in organochlorine pesticide GC-ECD chromatograms and it required right around 7 years to distinguish and affirm PCBs as ecological pollutants [41]. There are such countless PCB techniques right now being used that while contrasting information uncommon with find labs utilized a similar strategy. Early strategies, for example, EPA Technique 608 or 8082A utilized GC-ECD with PCB specialized combinations like Aroclors to adjust and evaluate results [71], but there are numerous limits to these strategies, which are talked about in Johnson et al.

In such cases it is critical to choose a technique that has the expected responsiveness but on the other hand can evaluate however many individual congeners as would be prudent to work on the possible force of the factual strategies utilized. In this occasion GCxGC is an exceptionally helpful device as it gives an additional element of detachment which fundamentally expands the settling limit. Utilizing GCxGC has took into consideration the recognizable proof of north of 190 of the 209 PCB congeners alongside concurrent ID of other organohalogenated toxins [49, 90]. This technique has been effectively applied to a few natural legal sciences applications, for example, deciding the provenance of ocean birds [87] and laying out the wellspring of PCB openness in people [32].

3.3 Assurance of Polychlorinated Dioxins and Furans

Despite the fact that dioxins, furans and PCBs are synthetically comparable, most dioxin and furan techniques are by and large considerably more exact and exact than most PCB strategies. Most of dioxin and furan strategies depend on US EPA 1613 and use isotope weakening with GC-HRMS to decide the seventeen 2,3,7,8-subbed harmful congeners. Due to the exceptionally harmful nature of these mixtures, they are managed worldwide and are in many cases identified in openness and pollution appraisals and ecological legal sciences examinations [114, 115]. Because of the broad example fixation and cleanup steps expected for dioxin strategies, technique times required to circle back are long and costs are fundamentally higher than those for different sorts of anlaytes [116, 117]. Nitty gritty polluted site portrayal for recovery can require an essentially huge number of tests to depict the debased region, which can be very expensive. Bioassays have been utilized, yet can require broad example separate cleanup to create precise outcomes. The QuEChERS system utilizes a changed extraction method to essentially diminish test planning time. Haimovici et al. ; fostered a strategy for site cleanup that essentially upgrades efficiency and diminishes logical completion time by a variable of at least 3 [114, 118].

3.4 Target investigation

There are a couple hundred synthetic substances that make up the POPs. These mixtures are normally resolved utilizing designated techniques [119]. Utilizing designated examination, a huge number of mixtures are neglected; this might be unfavorable in an ecological legal sciences examination where the wellspring of tainting is obscure. The advancement of season of flight mass spectrometers [106] and catching instruments (Orbitrap) [120] to increment awareness, examine speed and goal empowers them to be utilized for non-target full sweep examination. The utilization of examining instruments (TOF, Q-TOF, Orbitrap) empowers experts to acquire full sweep spectra which can be explored physically to get extraordinary mass to charge proportions and relegate natural sytheses for the suspect mixtures being researched [121, 122].

There are additionally a rising number of studies that have utilized non-designated strategies to recognize blended brominated/chlorinated fragrant mixtures, for example, dioxins, furans and PAHs in ecological examples [72]. Utilizing free non-endlessly designated strategies can assist with laying out additional precise degrees of hazard and give more demonstrative data to legal examinations. Mass spectra produced from complex examples can contain numerous thousand part particles. Mass imperfection plots (MDPs) can be utilized to handle this information and distinguished many mixtures in a solitary examination. The mass imperfection is the contrast between the ostensible and definite mass of a synthetic component or compound. Plotting the mass deformity against the mass to charge proportion permits comparative mixtures (homologues and congeners) to be distinguishe.

4 Conclusion:

Accurate POP analysis in environmental forensic investigations depends critically on sample preparation. Depending on the particulars of the material, a broad range of extraction techniques are used, including both conventional procedures like Soxhlet extraction and contemporary strategies like QuEChERS. These techniques seek to minimize target compound loss and maintain high recovery rates while successfully isolating the contaminants from their matrices. To ensure that the findings are reliable, it is essential to choose suitable standards and internal controls. To satisfy the growing needs of environmental forensic investigations, strict adherence to quality control norms and ongoing developments in sample preparation technology are required. This guarantees the continued accuracy, effectiveness, and legal justifiability of POPs analysis.

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