Review article

Review of scientific literature on available methods of assessing organochlorine pesticides in the environment

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ABSTRACT

Organochlorine pesticides (OCPs) are persistent organic pollutants (POPs) widely used in agriculture and industry, causing serious health and ecological consequences upon exposure. This review offers a thorough overview of OCPs analysis emphasizing the necessity of ongoing work to enhance the identification and monitoring of these POPs in environmental and human samples. The benefits and drawbacks of the various OCPs analysis techniques including gas chromatography-mass spectrometry (GC-MS), gas chromatography-electron capture detector (GC-ECD), and liquid chromatography-mass spectrometry (LC-MS) are discussed. Challenges associated with validation and optimization criteria, including accuracy, precision, limit of detection (LOD), and limit of quantitation (LOQ), must be met for a method to be regarded as accurate and reliable. Suitable quality control measures, such as method blanks and procedural blanks, are emphasized. The LOD and LOQ are critical quality control measure for efficient quantification of these compounds, and researchers have explored various techniques for their calculation. Matrix interference, solubility, volatility, and partition coefficient influence OCPs occurrences and are discussed in this review. Validation experiments, as stated by European Commission in document SANTE/11813/2017, showed that the acceptance criteria for method validation of OCP analytes include ≤20 % for high precision, and 70–120 % for recovery. This may ultimately be vital for determining the human health risk effects of exposure to OCP and for formulating sensible environmental and public health regulations.

1. Introduction

The class of persistent organic pollutants known as organochlorine pesticides (OCPs) has been utilized widely in public health and agricultural initiatives [1]. They are harmful chemical compounds that are used to combat various pests and weed development that is out of control; hence, impacting food security [2].

OCPs are endocrine disrupting chemicals (EDCs) with ability to interfere with hormonal homeostasis and the endocrine system, causes harm to humans and non-target organisms, act as teratogens, neuroendocrine disruptors, immunosuppressants, reproductive system inhibitors, and metabolic and lipid dysregulators, resulting in 3 million poisoning cases annually [3,4]. Health risks are significantly more likely to occur during the production and formulation of pesticides [5].

Due to their relatively high octanol-water partition coefficient, most OCPs are not easily soluble in water, but they are easily enriched in organisms and have the potential to bioaccumulate in the food chain, posing serious risks to ecosystems and public health

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[6,7]. Majority of OCPs were included in the Stockholm Convention since 2001. However, because of their ubiquitous nature, stability, volatility, resistance to degradation, and lipophilicity, OCPs have accumulated in both the natural environment and humans [4,8]. They enter the environment by infiltration, surface runoff, and volatilization, among other mechanisms [9].

More than half of all insecticides used worldwide comes from Asia. India is third in Asia after China and Turkey in terms of pesticide usage, ranking 12th globally. In 2018, India used over 58160 tonnes of pesticide, while consumption of pesticides in China, Japan, and the United States was about 13.07, 11.76, and 3.57 kg ha^{-1}, respectively [10].

Reliable and accurate analytical procedures are necessary to track their levels in environmental and clinical samples because of their persistent nature and potential health risks. In the creation of such analytical methods, method validation and optimization are vital processes [11]. Consistent analysis techniques can assist with regulatory compliance while preventing exposure to risky OCP levels [12]. A crucial phase in the validation and optimization process of OCP analysis is comparing the newly designed method to current methods [13]. It helps to make sure that the new approach is solid, dependable, and exact and that it can be used in a variety of labs and locations [14,15]. Quality control and quality assurance (QC/QA) systems aim to ensure that results have a high likelihood of being of acceptable quality and to decrease measurement errors to predetermined limits [16,17]. The reagents, testing utilities, equipment, operators, calibration procedures, and/or analysts vary, as well as the quality of the data. As a result, procedures for QC/QA were developed to guarantee the reliability of outcomes. Analytical processes must therefore make use of outcomes that have been validated. Users are interested in some indication of the caliber of the findings, which method validation demonstrates [18]. Although, this does not completely provide solutions to the discrepancy as there are still variations in the figures obtained from quality assurance. The most crucial parameters that researchers look for when evaluating technique validity are the limits of detection (LOD) and of quantification (LOQ) [19]. Others include accuracy (recovery), precision (relative standard deviation (RSD)), linearity, and external calibration [20]. It is not deemed necessary to investigate these effects separately. It is advised to adopt an experimental design (matrix) [21]. The analyte concentration in a sample matching to the lowest calibration point needed to satisfy analytical detection and quantitation criteria were referred to as method detection limits (MDLs) and method quantification limits (MQLs), respectively [22]. However, techniques for sample preparation can improve performance outcomes for improved recovery, higher sensitivity, and lower detection limits [18].

Several factors necessitate a critical evaluation of OCP technique validation and optimization in environmental and clinical samples. Despite banning of OCPs in early 1970s, some of them including DDT, HCH, and HCB are still in use in the global South, with renewed interest in measuring levels in different environments, e.g., marine, freshwater, and terrestrial [23–25]. Both atmospheric deposition and the adulteration of pesticides that are not prohibited can be attributed to the existence of the above mentioned OCPs. The lack of sufficient resources and infrastructure to support research and analytical facilities is a significant challenge faced in Africa and developing countries, making it difficult to collect, process, and analyze samples accurately. This can result in inaccurate assessments of OCP levels in the environment and clinical samples due to the absence of required instruments, chemicals, and standards. To ensure sensitivity, accuracy, and precision, complex analytical procedures such as solid-phase microextraction (SPME), gas chromatography-mass spectrometry (GC-MS), and high-performance liquid chromatography (HPLC) may be necessary, which are expensive and require skilled personnel. It is also necessary to establish procedures for identifying and quantifying OCPs to avoid inconsistent results and ensure data comparability. Furthermore, conducting a critical evaluation can identify areas for progress and suggest new lines of investigation since the creation and verification of analytical techniques are ongoing processes. Lastly, multiple techniques are available for investigating OCPs, each with its advantages and limitations. Therefore, it is essential to critically evaluate and choose the best method for a particular sample type and analyte.

Literature reports have demonstrated that accuracy and precision are construed in a variety of ways while still pursuing the same objective [26–31]. As a result, various calculations may yield different outcomes. The purpose of the present article is to: (1) critically assess the existing literature and methodologies; (2) identify inconsistencies and limitations; (3) provide insights into the best practices for accurate and reliable analysis of organochlorine pesticides; and (4) provide insights into the implications of these discrepancies on the accuracy, reliability, and comparability of the obtained results.

2. Methodology: literature review

Online internet database was searched to perform a systematic evaluation of OCP validation methodologies. The Clarivate Web of Science search string was used, and the reviewed database includes studies published between 2020 and March 2023, with more than half published in 2020. The search phrases "organochlorine pesticides" and "OCPs" were used. Studies which did not report the quality assurance were discarded, although google scholar was consulted to elaborate on the reviewed studies. The final search of the articles for this review was conducted on December 21, 2022. A total of 620 articles were retrieved from the web of science. All terms were joined by "OR". Though review papers were retrieved, they were not included. All the papers' abstracts, conclusion, and methodology were screened to decide the final eligible articles. The language of included articles was limited to English.

3. Result and discussions: literature review

3.1. Extractions and cleanup

Several authors have discussed extensively on the extraction and cleanup methods of different pesticides, detection techniques, their advantages and disadvantages from various matrices, as well as their LODs [32,33]. The utilization of solid-phase extraction (SPE) [34], liquid-liquid extraction (LLE) using silica-alumina columns for purification [35], quick component separation, and reliable

Surface-enhanced Raman Scattering (SERS) [36], are just a few of the extraction techniques for water samples that have been documented. Although sample complexity, trace content, and poor molecule affinity for metal surfaces provide significant hurdles for SERS [36]. OCPs have been extracted from sediment using the soxhlet method [37–39]. Isotope dilution and centrifugation [40,41] and SPE [42,43] have been utilized for human serum extraction, while salting-out liquid-liquid extraction (SALLE) combined with dispersive liquid-liquid microextraction (DLLME) has been reported for human tissue (liver, kidney, brain, etc) [44].

These procedures call for moderate to large quantities of potentially dangerous organic solvents, which is time consuming. Therefore, more sophisticated methods using little or no solvent, such as solid-phase extraction, solid-phase microextraction, and dispersive liquid-liquid microextraction (DLLME), have been proposed to decrease the amount of organic chemical residues and to raise the concentration factors [45,46], and also produce data with a lower detection limit [47]. Ultrasonication for maize [48], pressurized solvent extraction for egg and Soxhlet extraction with column cleanup have been reported for fruits and vegetables, cheese, yoghurt, fish, meat, cereal, pulses, maize and air samples [49–51]. The Quick Easy Cheap Effective Rugged and Safe (QuEChERS) [52], solid phase microextraction (SPME) [13], and soxhlet extraction have all been used to remove OCPs from fish samples. The multiclass or multiresidues analysis of various pesticide residues in fruits and vegetables has made extensive use of QuEChERS [53]. Given its benefits (high recovery of pesticides, need for very little labware, use of smaller amounts of organic solvent and the use of non-chlorinated solvents, high sample throughput, and increased safety for laboratory workers), QuEChERS method has recently attracted attention for pesticides analysis [54].

The effectiveness of the extraction and purification procedure are influenced by solubility and depends on how well OCPs dissolve in the solvent utilized. Some of the well-known and extensively utilized solvents used in the extraction process include dichloro-methane, hexane, methane, acetone, diethyl ether, toluene, among others [55]. Based on solubility factors such solvent polarity, dispersion coefficient, and hydrogen bonding, these solvents' extraction effectiveness is determined. Large amounts of OCPs may be extracted using hexane/methanol in a 4:1 (v/v) ratio and hexane/acetone in a 1:1 (v/v) ratio [55,56]. The acetonitrile/ethyl acetate solvent mixture was used in a study because the targeted pesticides are polar, and recovery of pesticide residues was investigated using various proportions (v/v) of acetonitrile and ethyl acetate [57].

This strategy is to use extraction and purification techniques tailored to the target compounds to selectively identify and clean the target OCPs from the sample matrix. OCPs were selectively extracted from mango matrices using dispersive solid-phase extraction (dSPE) or liquid-liquid extraction (LLE) [57]. For fish sample extraction, a combination of polar and non-polar solvents has often proved more effective than a polar solvent [55,58]. An appropriate extraction solvent (1-decanol) with acceptable analyte solubility and water immiscibility revealed good accuracy, relative recovery, and low LOD values [59]. A recently developed solvent-free analytical approach called solid-phase microextraction (SPME) offers the advantages of being straightforward, having lower detection limits, and having good reproducibility [60].

Table 1
Procedures for recovery studies and RSD.

S/N	Recovery	RSD injection replication	Reference
1	Procedural and spike blank		[83],
2	Spiked sample	5 injections	[84]
3	Method and sample blank		[77]
4	Procedural and spikes blank, duplicate samples		[85]
5	Blank and real sample	7 replicates	[26]
6	Method and blank sample		[86]
7	Field and equipment blank		[87]
8	Matrix spike duplicate, and matrix spike triplicate		[52]
9	Surrogate recovery		[88,89]
10	Spiked blank sample	7 replicates	[90,91]
11	Solvent blank, a matrix blank and a matrix spike and its duplicate		[92]
12	Spiked blank samples		[93]
13	Field and method blank		[94]
14	Field and lab blank	10 replicates	[95]
15	Solvent, field, and procedural blank		[66]
16	Procedural, matrix and sample blank		[96]
17	Spiked sample	5 replicates	[19]
18	Field blank		[51]
19	Field and procedural blank	3 replicates	[97]
20	Procedural blank		[31,98]
21	Method blank, parallel samples, blank solvent recovery surrogate, internal standard, QC standard sample		[99]
22	Blank sample		[100]
23		6 replicates	[101]
24		7 replicates	[102]
25	Blank matrix	7 replicates	[103]
26	Field blank		[104]
27	Duplicate samples, blank and spike samples, CRM		[105]

Table 2

Quality control/assurance in Environmental matrices.

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m³)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m³)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
River	7	2×10^{-6}–5.1×10^{-5}	1×10^{-5}–$1.^{69x10-4}$	73.3–115.5	4.5–14.6	GC-MS. "splitless mode with initial column flow rate of 1.9 mL min− 1. Initial temperature of oven at 60 °C for 3 min, 110 °C (2 min) at the rate of 5 °C min− 1, reaching 200 °C at 8 °C min− 1 and finally attaining the temperature of 315 °C at 5 °C min− 1 with final holding time of 10 min"	[106]
River water	14			66–80		GC-MS/MS: "The initial temperature of oven was set at 150 °C for 3 min, and then raised to 290 °C at a rate of 4 °C/min and held for 10 min"	[66]
River water	8	0.001–0.005		81.6–109		GC-ECD: Split. "the column temperature starts at 160 °C with a hold time of 2 min and increased to 270 °C at a rate of 4 °C·min⁻¹ with a 2-min hold. Tail gas flow was 60 mL/min"	[34]
Snow	13	$1.^{1x10-6}$–$2.^{6x10-5}$	$3.^{2x10-6}$–$7.^{8x10-5}$	CRM: (27–137 Internal standard: 70–98	10–25	GC-MS: "Initial temperature of oven at 40 °C for 2 min, 150 °C (0 min) at the rate of 25 °C min− 1, reaching 200 °C at 0 °C min− 1, 280 °C at 10 °C min− 1 and finally attaining the temperature of 300 °C at 5 °C min− 1 with final holding time of 5 min" RT 12.90–13.90	[80]
Glacier-ice core	25	4×10^{-6} -1.53×10^{-4}		9.67–112.4		GC-MS: splitless, "temperature program: 80 °C (2.00 min) to 110 °C at 7 °C min-1 then 3 °C min-1 to 250 °C and a final ramp of 10 °C min-1 to 285 °C with a hold of 5 min" ECDs: "temperature program: 80 °C for 2.00 min, then 10 °C min-1 to 150 °C then 2 °C min-1 to 280 °C hold for 5 min. Hydrogen was used as a carrier at 1.0 mL min-1"	[81]
Water wells	20	0.02–0.74	0.05–2.46			GC-MS: "the initial temperature of 90 °C (hold 1.3 min), increased to 125 °C at 15 °C/min, 165 °C at 5 °C/min, 195 °C at 2.5 °C/min, and finally, it was increased to 280 °C at 20 °C/min (hold for 4 min)". RT 18.38–30.59	[107]
farmland, river, and fishpond water	4	0.46–0.97 nM[a]		90.20–109.4	4.23	GC-MS	[36]
Seawater	10	0.0001–0.0004		Standard spike:20–115; surrogate: 66-84		GC-MS/GC-ECD	[35]
Seawater and sediment	50	1×10^{-5}–5.6×10^{-4} (seawater) 0.01–1.04 (sediment)		69.5–118.2; 75.2–120.4	9.5 (water); 8 (sediment)	GC-MS	[108]
Surface water and Sediment	18	Water: 9×10^{-6}–2.5×10^{-4}; sediment: 0.018–0.500		Water: 690–122; Sediment: 77-108		GC-MS/MS: Splitless. "the GC temperature programs were as follows: 40 °C for 1 min; 40 °C–120 °C (40 °C min⁻¹), held for 0	[109]

(*continued on next page*)

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Water, sediment	22	IDL:2.9–112.2 pg[a]; MDL: 5-227		22.42 %	0.75–57.2 %	min; 120 °C–240 °C (5 °C min^{-1}), held for 0 min; 240 °C–300 °C (12 °C min^{-1})" "Gradient temp: 80 °C for 0.75 min, 40.0 °C/min to 170 °C, 2.5 °C/min to 236 °C, then 40.0 °C/min to 275 °C, hold 9.62 min. Run time: 40.0 min. 1 µL injection"	[82]
Water and sediment	11	Water: 0.2–0.4; sediment: 1-3	Water: 0.7–1.3; sediment: 4-15	Water: 94–98; sediment:94-103		GC-ECD: "The column temperature was programmed from 90 °C (3 min) at a rate of 25 °C min to 200 °C, fixed for 15 min and then at a charge per unit of 30 °C min to 265 °C held for 5 min to 275 °C (15 min) at 3 ° C min^{-1}".	[110]
Sediment, river water	23		MDL:RW: 2.6 × 10^{-7} –9.2 × 10^{-6}; sed: 1.8 × 10^{-4}–7.6 × 10^{-4}; 1.5 × 10^{-4}–4.1 × 10^{-4}	RW 62.8–86.3; sed: 66.4–84.3; muscle: 73.1–105	<15	GC-HRMS: "The oven program was 100 °C (1 min), 20 °C/min to 200 °C, and 2.5 °C/min to 270 °C (5 min)"	[111]
Water, sediment, benthos, phytoplankton	20	MDLs: Water: 1 × 10^{-5}–2.57 × 10^{-3}; sediment: 0.01–2.81		80–112	<5	GC-µECD	[112]
Surface water	20	1 × 10^{-6}-7.2 × 10^{-5}		Surrogate recovery: 88–117 and 85–110		GC-ECD: Splitless, "the initial temperature of 120 °C was ramped to a final temperature of 290 °C with a hold time of 3.5 min"	[113]
Water	10	MDL: 0.00010 to 0.00040		20 to 115; surrogate: 66 to 84			[114]
water	19	250–3400		89–105	3.9–13.2	GC-MS: "an initial temperature of 50 °C, which was then to 180 °C at 20 °C/min and held for 1.5 min. This was then increased to 200 °C 3 °C/min and held for 3 min. Finally, it was increased to 230 °C at 5 °C/min and held for 5 min. The total run time was 28.667 min"	[115]
water	19	0.00001–0.002		87–105		GC-ECD, GC-MS: Splitless, "the column temperature was programmed from 60 °C to 170 °C at 10 °C/min, a hold for 2 min, followed by an increase to 280 °C at 5 °C/min, and a hold for 3 min, and finally at 15 °C/min to 300 °C"	
Water	14	IDL: 0.001–0.116	IQL: 3.6–388	67.3–129	9.2–18.1	HPLC-MS/MS: "The gradient elution program was as follows: 0–0.5 min, 10 % B; 3.0 min, 90 % B; 11.0 min, 90 % B; 11.1 min, 10 % B; 11.1–15.0 min, 10 % B"	[116]
River and pond water	6	0.000011–0.000058	0.00009–0.0.00193	River: 85.9–122; pond: 82.9–96.2	2.7–14.5	GC-MS: Splitless, "the initial oven temperature was at 80 °C and held for 0.5 min, increased to 200 °C at a rate of 30 °C/min, then increased to 250 °C at a rate of 20 °C/min and held for 2 min, and finally	[117]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Agricultural effluents, WWTP	20	0.0001–0.005	0.0005–0.01			increased to 300 °C a rate of 25 °C/min and held for 2 min". RT 13 min GC-MS: "the initial temperature of 80 °C (hold 1 min) increased to 170 °C at 10 °C/min (hold 10 min) then increased to 230 °C at 4 °C/min and finally increased to 280 °C at 3 °C/min (hold for 2 min), with a total acquisition time of 41 min"	[118]
Water, DP, SPM	25	MDLs: 0.00021 to 0.00152	MQLs: 0.00070 to 0.00507	surrogate spiked: 61.3–108.6		GC-MS: RT. 13.47–21.72 min	[119]
Surface and ground water	7	0.00008–0.00018		78–91	2–6	GC-MS: "The oven temperature began at 80 °C–200 °C at a rate of 10 °C per minute (5 min hold time) and increased to 270 °C at a rate of 5 °C per min (5 min hold time)"	[120]
Surface and groundwater	8	0.00003–0.00018		85–92.7	3.9–11.2	GC-ECD: "The oven temperature began at 60C for 1 min and increased to 290C (10 min hold time) at a rate of 4 C/min"	[121]
Virtual Organism, sediment	30		VO: 0.0043–0.0435; sediment: 0.00039–0.0276	VO: 22–72; sediment: 19-90	VO: 13–39; sediment: 12-40	HRGC/HRMS	[27]
Water and sediment	18	Water: 8.8 to 16.4; sediment: 0.0219 to 0.0298	water: 0.0279 to 0.0497 sediment: 0.0698 to 0.0902	Water: 74–110; sediment: 91–125	0.9–1.8	GC-MS: Splitless, "the oven temperature was set at 150 °C and held for 2 min, raised to 270 °C at 14 °C/min and held for a further 2 min"	[38]
Water and sediment	30	Water: 0.00003–0.0001; sediments: 0.00050–0.001		Water: 88.2 to 103.1; sediment: 72.7 to 95.5		GC-MS: "The oven temperature of the GC was initially held at 70 °C for 5 min and raised to 280 °C at 4 °C/min and kept for 5 min""	[39]
WATER AND SOIL	26	WATER: MDLs: 0.00001–0.00089; SOIL: 10-980		Water: 41–113 soil: 57-121		GC−MS/MS: "The oven temperature was programmed as follows: the initial temperature (50 °C) was held for 1 min and increased by 25 °C/min to 125 °C and then increased by 10 °C/min to 300 °C and held for 2 min"	[122]
Water sample	5	240–330	740–980		1.4–3.5	GC-ECD: "The oven temperature was set as follows: initial oven temperature of 170 °C for 2 min, increasing to 300 °C at a rate of 10 °C/min". RT 7.45–13.26	[123]
Water	8			Mixed reference sample: 70–140; surrogate: 70-130	<5	GC-MS: Splitless,. "the initial oven temperature was 80 °C, which was maintained for 1 min, then raised to 160 °C at a rate of 30 °C/min, maintained for 1 min, and finally raised to 265 °C at a rate of 3 °C/min and maintained for 1 min"	[124]
Water	24	MDL: 0.00001 to 0.00002		Spike mean: 66; surrogate mean: 92	<20	GC-MS; GC-ECD	[99]
Water	8	0.0024–0.0462		77.7–106.3		GC-MS: "oven temperature program began at 100 °C (held for 0.5 min) and was raised to 175 °C at 40 °C·min^{-1} (held for 2 min),	[125]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						before ramping to 190 °C at 5 °C·min^{-1}. It was then further increased to 205 °C at 20 °C·min^{-1}"	
Surface water	13	MDLs: 0.000022–0.000069		Surrogate: 80–110	1.50–5.54	GC-ECD: "initially at 80 °C (equilibrium time 1 min), increased to 150 °C at the rate of 20 °C/min and held for 2 min, before reaching at 300 °C at the rate of 5 °C/min, and then held for 5 min"	[126]
Surface water	9	0.000002–0.00003	0.000003–0.000061	Int std: 72-93	<1.8	GC-MSD: "The column temperature was initially held at 50 °C for 1 min, then increased to 160 °C at 7 °C/min and finally increased to 240 °C at 10 °C/min, and was held for 18 min"	[127]
Surface water	14		Water: 100; SM: 0.1	92–120		GC-MS	[128]
Water lake	20	MDL: 2.81 e−6-1.58e-4	MQL: 2.81e-06-0.000158		1.4–11.9	EC-ECD	[129]
Surface water	7		0.0006–0.003	80–115	8–15	GC-ECD: Split, "temperature program of hold for 1 min at 100 °C, drop the temperature at 10 °C/min to 240 °C and hold for 1 min, drop at 1 °C/min to 260 °C and hold 1 min and finally drop at 10 °C/min to 300 °C and hold 10 min"	[130]
Surface water	13	0.000006–0.00002		82–124	<20	GC-ECD: "Oven temperature (Initial) 100 °C (1 min) → 180 °C at 25 °C/min (2 min), ramp 5 °C/min → 240 °C (1 min), and at 4.5 °C/min to 260 °C (2 min) → at 10 °C/min to 280 °C (5 min). RT: 8.839–23.195 min"	[131]
Water	8		**0.001–0.01**	94.5–106.8		GC-MS: "initial temperature of 80 °C which was maintained for 1 min, followed by an increase in temperature to 230 °C at a rate of 10 °C min^{-1}, which was then maintained for another 4 min"	[132]
water		0.00028–0.01655	0.00092–0.05516	63.6–125	1.03–17	GC-MS	[133]
Water	4	40–97	12–29	91.8–103.5	2.7–4.5	MSPE-HPLC-UV: "The column temperature was set at 30 °C and the detection wavelength was set at 238 nm"	[134]
Water	51	MDLS:0.0001–0.0031		75.2–105.8	1.8–11.30	GC-MS	[135]
water	10	0.00006–0.003	0.0002–0.01	91–109	3–10	GC-MS: "The oven was programmed at 80 °C and increased to 290 °C at 20 °C/min and held at 290 °C for 4.75 min"	[45]
Lagoon water	7			51–120		GC-ECD: "The initial oven temperature is 60 °C (for 2-min hold), raised to 160 °C at 20 °C/min then 200 °C (for 10-min hold), and was maintained at 250 °C for 2 min"	[136]
Drinking eater	5	305–2340		87–92	4.16–6.06 %	"The oven temperature was programmed with an initial isothermal at 150 °C held for 2 min, which was then increased to 210 °C at 10 °C/min, and was held for 1 min, to increase the temperature to the	[137]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						final value of 300 °C at 5 °C/min and held for 5 min"	
Drinking water	6	10–20	40–200			GC-MS:"The oven temperature was set at 80C and kept at this temperature for 1 min. Then, the temperature was increased to 175C with a ramp of 30C/min and then kept for 4 min. Next, the oven temperature was increased to 225C with a ramp of 3C/min and kept at this final temperature for 10 min"	[138]
Tap water	26	7e-07-0.00009		Spiked: 72–122; internal std: 84-108	<4	GC-MS: "80 °C for 1 min; 80 °C-150 °C (20 °C/min); 150 °C-287.5 °C (5 °C/min); with temperatures of 150 °C, 230 °C, and 280 °C for the quadrupole, ion source, and interface, respectively"	[139]
Surface water, SPM	14		MQLs: 2.5e-05–5e-05	Water: 63–127; SPM: 72-126	<5	GC-ECD: Splitless, "oven heating started at 90 °C and 1min hold, ramped to 210 °C at the rate of 10 °C/min with 1min hold, and then to 230 °C at 1 °C/min with 10min hold, and finally, it increased to 250 °C at 1 °C/min"	[140]
Water, SPM	26	0.00024–0.00183	0.0008–0.00614	SF: 62.5–126; SPM: 60.8–114.8		GC-MS: "The oven temperature was programmed as follows: initially at 70 °C (equilibrium time 1.5 min), increased to 200 °C at the rate of 10 °C/min, continually increased before reaching at 320 °C at the rate of 7 °C/min, and then held for 3 min"	[7]WWEE
Water, WTP	6	25–512	800–1630			GC-MS: "The oven temperature was started at 80 °C and kept at this temperature for 1 min. Then, the temperature was increased to 175 °C with a ramp of 30 °C/min and then kept for 4 min. Next, the oven temperature was increased to 225 °C with a ramp of 3 °C/min and kept at this final temperature for 10 min"	[141]
Water, sediment	25	Water: 6.3–21; sediment: 1.3–4.1	Water: 21–68 ng/mL; sediment: 4.2–14	CRM: 79.2 to 117; Water: 46.6 to 119; sediment: 86.7–117	Water: 0.05–20; sediment: 0.7–17	GC-MS: "initial temperature of 50 °C, held for 1 min, ramped at 5 °C/min to 180 °C, held for 3 min, and increased to 300 °C at 10 °C/min, maintained for 7 min, equating to a total run time of 49 min"	[71]
Water, Soil, sediment	20			Surrogate: 65 and 105	<15	GC-ECD: "The oven temperature program began at 100 °C, held for 1 min, raised to 200 °C at 4 °C·min^{-1}, then to 230 °C at 2 °C·min^{-1}, and ramped to 280 °C at 8 °C·min^{-1}, and held for 15 min"	[142]
Water, planktons and corals	8	MDL: Seawater: 2 e−09-1.6e-08: marine biota; 0.0009–0.0013		74–110		GC-MS/MS: "The oven temperature was programmed as follows: 80 °C for 5min, at 20 °C min-1 to 160 °C, at 4 °C min −1 to	[89]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m³)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m³)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						240 °C, at 10 °C min −1 to 295 °C with a final hold for 2min". RT: 11.4–26.3	
Water, biota	16	MDL: 3.3e -07-1.25 e−06		75–89	5.9		[143]
lake	12	0.01		40–100		GC-MS/MS: "The GC programming was as: The primary temperature was 70 °C and hold for 2 min, then ramped up to 150 °C at 25 °C/min, then ramped up to 200 °C at 3 °C/min, finally ramped up to 280 °C at 8 °C/min and hold for 10 min"	[144]
Gas and particulate phase	13	0.044–0.145		74.6, 89.6		GC-MS/MS: "The oven temperature program was as follows: initial temperature of 100 °C (hold for 1min), heat to 220 °C at a rate of 5 °C/min (hold for 10min), then heat to 280 °C at a rate of 4 °C/min and hold for 5min"	[145]
SOIL	10	0.012–0.061		78.80–110.48	<10	GC-ECD: "The column oven-heating program was as follows: initial temperature of 80 °C, ramped up to 260 °C at the rate of 10 °C·min− 1, then heated to 280 °C at the rate of 20 °C·min− 1, and held for 5 min"	[146]
Soil	8	0.012–0.061		78.80–110.48	<10	GC-ECD: "The oven temperature of gas chromatography was set to start at 120 °C, ramped to 260 °C at the rate of 10 °C·min⁻¹, and then to 280 °C at a rate of 20 °C·min⁻¹, and finally held for 5 min"	[147]
Soil	23	MDL: 0.004–0.360		53.9–112	1.6–8.5	GC-HRMS: Splitless, "the oven temperature program was as follows: 100 °C (1 min) → 20 °C/min → 180 °C (5 min) → 0.5 °C/min → 186.5 °C (20 min) → 10 °C/min → 250 °C (24 min) → 40 °C/min → 300 °C (30 min)"	[90]
Soil	24	0.00011–0.00549		Surrogate: 60-107	<10	GC-MS/MS: "The temperature program of the GC oven was initially set at 100 °C, then increase to 200 °C at the rate of 10 °C/min, keep heating to 230 °C at the rate of 1 °C/min with the holding time of 1 min, and finally reach 290 °C at the rate of 10 °C/min with the holding time of 10 min"	[94]
Soil	19			86–135		GC/HR-MS: "The GC temperature was programmed as follows: 60 °C was held for 1.5 min, then increased to 140 °C at a rate of 10 °C/min, then to 220 °C at a rate of 4 °C/min, 250 °C at a rate of 2 °C/min, and finally to 300 °C at a rate of 8 °C/min". "Chromatographic analysis was performed at the following temperatures: 120 °C for 1 min and then increased to 180 °C at a rate	[95]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						of 20 °C/min, then to 260 °C at a rate of 2 °C/min, and finally to 300 °C at a rate of 5 °C/min for 4 min"	
Soil	3	0.022–0.024		97.8–111.3	7.1–13.1	GC-MS: Splitless. "the column temperature was at 80 °C for 1 min, increased to 150 °C at a rate of 20 °C/min, then increased to 210 °C at a rate of 5 °C/min, and finally increased to 280 °C at a rate of 10 °C/min; thereafter, the temperature remained constant for 5 min"	[148]
Soil	15	500–2000	1000–2x10^4			GC-MS: Splitless, "the oven temperature was programmed starting at 45 °C and held 0.5 min, followed by an increase of 30 °C min^{-1} up to 90 °C, held 0.5 min, then 10 °C min^{-1} up to 310 °C for 3 min"	[149]
Soil	28	0.036–0.183				GC-ECD: "the oven temperature began at 100 °C (equilibrium time 1 min), rose to 200 °C at 4 °C/min, then to 230 °C at 2 °C/min, and at last reached 280 °C at a rate of 8 °C/min, held for 15 min"	[150]
Soil	25	0.018–0.058	0.062–0.199	Surrogate: 59.2–124		GC-ECD: "The oven temperature was set to start from 100 °C (held for 1 min), and then increased by a rate of 4 °C/min to 200 °C, by a rate of 2 °C/min to 230 °C, and at last by a rate of 8 °C/min to 280 °C, with a final holding time of 15 min"	[151]
Soil	25	MDL: 0.001–0.02		74.8 and 86.9	<20 %;	GC-ECD/GC-MS: "initially 100 °C for 1 min, 4 °C/min to 200 °C, 2 °C/min to 230 °C, and 8 °C/min to 280 °C for 15 min"	[152]
Soil	26	MDLs: 0.002–0.008		72.8–100.3	1.5–7.7	HRGC-HRMS: Splitless, "120 °C (2 min hold)-20 °C/min to 200 °C (2 min hold)-5 °C/min to 240 °C (2 min hold) −2.5 °C/min to 270 °C (2 min hold)-5 °C/min to 300 °C (2 min hold)." Total 35 min"	[153]
Soil	20	0.05–0.5	0.16–1.55	80.3–109	<10	GC-MS: "The initial oven temperature was maintained at 100 °C for 2 min, ramp to 180 °C at a rate of 15 °C/min, and raised to 300 °C at a flow rate of 3 °C/min and held for 9 min"	[154]
Soil	18	MISOLEX: 0.005–1.6; QuEChERS: 0.001–1.48	MISOLEX: 0.02–3.85; QuEChERS: 0.04–4.93	MISOLEX: 65.8–180.9; QuEChERS: 67.8–169.3	MISOLEX: 1.3–22.4; QuEChERS: 1.2–40.9	HS–SPME–GC–MS: SIM, "the initial oven temperature was set to 90 °C and held for 3 min. The temperature was ramped to 150 °C at a rate of 15 °C·min^{-1}. Then, it was ramped to 280 °C at a rate of 5 °C·min^{-1} and held for 3 min. RT 18.99–33.44 min"	[75]
Soil	213 Pesticides; 12 OCPs			OCPs: 69–140; Pesticides: 21–197.3	OCPs: 8.2–41.6;	LC-MS: "The sheath gas was set at 12 L min^{-1} at 330 °C. The desolvation and nebulizing gas temperature was 190 °C	[76]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
					pesticides: 0.8–63.6	and the flow rate was 11 L min^{-1} with a pressure of 26 psi. The capillary voltages were set at 3900 and 2600 V in positive and negative ionization mode, respectively. The cycle time was 700 ms and dwell time 3–83 min; GC-MS: Splitless, "The column temperature was maintained at 80 °C for 1.8 min, increased to 170 °C at a rate of 40 °C/min, then increased to 310 °C at a rate of 10 °C/min and held for 3 min"	
Soil	12	0.048 and 3.125	0.5 and 20	67.9–119.6	3.1–31.6	GC-MS: "The temperature ramp was programmed as follows: (a) 80 ∘C—1.8 min; (b) 80 °C to 170 °C at a rate of 40 °C min−1; (c) 170 °C to 310 °C at a rate of 10 °C min−1; (d) 310 °C for 3 min. The total time for each analysis was 20.75 min"	[15]
Soil	8	DL: 0.002–0.05	QL: 0.158–0.169	80 to 110	5–10	GC-ECD: "The oven temperature of gas chromatography was set to start at 120 °C for 1 min, ramped to 240 °C at the rate of 7 °C/min and held for 5 min. RT: 9.07–16.912 min"	[155]
Soil	10			50–120		GC-ECD: "initial oven temperature was 100 °C (equilibrium time 1 min), raised to 200 °C at a rate of 4 °C·min^{-1}, then increased to 230 °C at 2 °C·min^{-1}, then raised to 280 °C at 8 °C·min^{-1} and held for 15 min"	[156]
Soil	14	0.01		81–118		GC-MS	[157]
Soil		0.03–0.20		90.1–93.7		GC-MS: Splitless, "the initial oven temperature was maintained at 100 °C for 2 min, ramp to 180 °C at a rate of 15 °C/min and then raises to 300 °C at a flow rate of 3 °C/min and held for 9 min"	[158]
Soil	OCPs (23), CUPs (24)	0.001–7.83	0.003–26.1	Procedural blank: 70-130 Spiked blank: 71.3–126	<20	GC-TQ-MS: Splitless, "held in 1 min at 60 °C, then ramped to 120 °C at the rate of 40 °C/min, and finally up to 310 °C with 5 °C/min and held for 3 min"	[83]
Soil	8	4–10	2.4–32	75–10	0.1–4.2	GC-MS: "The temperature of 80 °C lasted for 2 min, then raised to 210 °C (10 °C/min), and remained constant for 10 min, and further raised to 250 °C (10 °C/min) and remained constant for 5 min (PCBs and OCPs)"	[159]
Soil	4	0.002–0.003		75–105	<10	GC-ECD: "The initial temperature was held at 100 °C (equilibration time of 1 min) and raised to 200 °C at 4 °C/min, then to 230 °C at 2 °C/min, and finally raised to	[160]

(*continued on next page*)

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Soil	20	0.1		72.5–124.1	1–6.7	280 °C at a rate of 8 °C/min, and held for 15 min" GC-ECD: "The oven temperature was set at 80 °C initially for 2 min, and increased to 170 °C at the rate of 25 °C/min, then ramped to 250 °C at 5 °C/min and held for 2 min, and then raised to 280 °C at 25 °C/min and eventually held for 2 min"	[161]
Soil	23	0.04–1.21		63.9 and 112.1	0.4 and 26.2	GC-MS/MS	[162]
Soil	14			98–102		GC-MS/MS: "temperature program 100 °C, 1 min; 10 °C/min to 160 °C, 4 min and 10 °C/min to 260 °C, 2 min, transfer line temperature 280 °C, total analysis time 19.2 min"	[163]
Soil	8	0.0045–0.034		78–107	3.0–6.9	GC-QTOF-MS: Splitless, "the GC oven temperature profile was started at 100 °C and maintained at this temperature for 3 min, then it was ramped to 170 °C at a rate of 5 °C/min, subsequently ramped to 300 °C at a rate of 10 °C/min, and then the back-flush was performed for 0.5 min at 310 °C"	[164]
soil		0.02–0.15				GC-ECD: Split, "initial run temperature 80 °C, ramped at 50 °C/min to 210 °C, then ramped at 1 °C/min to 220 °C and held for 1 min, and finally ramped at 2 °C/min to 230 °C"	[165]
soil	14	IDL: 0.001–0.013		63–127		GC-ECD: "Increase the temperature to 80 °C during 1 min, then increase the temperature to 150 °C by the rate of 15 °C/min, up to 250 °C with 5 °C/min, and eventually up to 300 °C during 5 min"	[166]
Soil	4	0.628–3.68	2.093–12.27	81.42–110.7	1.68–9.43	GC-ECD: Split, column temperature: 250 °C"	[167]
Soil	6	0.10		86–90	3–10	ECD: "0 °C for 1 min, 30 °C/min to 180 °C, 180 °C, for 1 min, 3 °C/min to 205 °C, 205 °C for 4 min, 20 °C/min to 290 °C, 290 °C for 7 min"; GC-MS/MS: "70 °C for 2 min 25 °C/min to 150 °C for 0 min; 3 °C/min to 200 °C for 0 min; 8 °C/min to 280 °C for 10 min hold time"	[168]
Topsoil	(8 OCPs and chlorpyrifos; 32 CUPs	UPLC-MS/MS: 0.002–0.23; GC–MS: 0.1–1.2	UPLC-MS/MS: 0.02–0.75; GC–MS: 0.3–4.3	76.7–110.3	1.7–11.8	GC-MS, UPLC-MS/MS	[169]
Soil, moss	7	0.006–0.032		76–114	<4	GC-ECD: "The column temperature was from 90 °C (with 1 min hold) to up to 180 °C at 30 °C/min (1 min hold), and then up to 240 °C at 2 °C/min with a hold of 20	[170]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						min and finally up to 260 °C at 5 °C/min with an 8 min hold"	
Surface soil	20	0.001–0.0028		85–110		GC-MS: "Oven temperature for GC-MS analysis was set at 60 °C for 1 min and increased to 290 °C (10 min hold time) at the rate of 4 °C/min"	[90]
soil, spring water, river water, spring sediment, and river sediment	24	MDL: water: 1×10^{-5}-2x10^{-5} sediment/soil: 1×10^{-5}-2x10^{-5}		Surrogate: 70.8 and 86.6	<20	GC-ECD: "The GC oven temperature program was set as: initially 100 °C for 1 min, 4 °C/min to 200 °C, 2 °C/min to 230 °C, and 8 °C/min to 280 °C for 15 min"	[171]
Air, soil water, sediment	Endodulphan	MDL: Air: 2.5×10^{-4}-5.8 $\times 10^{-4}$ soil & sediment: 2.95×10^{-3}-5.59 $\times 10^{-3}$; water: 4.34 $\times 10^{-6}$ -3.02 $\times 10^{-5}$		Air:51–112, soil: 57–102, water: 57–105 sediment: 53–107		GC-HRMS: "Splitless, the initial oven temperature was set at 80 °C, raised to 150 °C at 20 °C/min, then to 235 °C at 5 °C/min, and finally to 300 °C at 15 °C/min, where it was held for 5 min"	[91]
Dust	OCPs			77 and 95		GC-MS: "The initial temperature of the column chamber was 60 °C and held for 1 min, then increased at a rate of 4 °C/min to 280 °C and held for 10 min, then increased at a rate of 10 °C/min to 300 °C and held for 10 min"	[172]
Dust	20	0.06–0.38	0.06–0.38	71–112	2–17	GC-MS/MS: "The GC oven temperature program was set as follows: held at 70 °C for 5 min, ramped at 10 °C/min to 160 °C and then at 5 °C/min to 280 °C, held for 5 min, ramped at 20 °C/min to 300 °C, and finally held for 5 min"	[173]
Indoor dust	16	MDL: 0.004–0.2		Surrogate: 46.4–120.51		GC-MS: "initial oven temperature was maintained at 100 °C at the rate of 20 °C/min and later increased to 240 °C and 296 °C at the rate of 4 °C/min and 8 °C/min"	[5]
Indoor dust	26	MDL: 0.00131–0.0073		Surrogate: 88 to 110 and 99–121		GC-MS: "The GC oven program used was: 60 °C for 1 min, 30 °C/min to 220 °C held for 0 min, and 5 °C/min to 300 °C held for 15 min"	[174]
Sediment	11	0.002–042	0.007–0.141	78.46–98.47		GC/MS-MS TSQ: "The column temperature started at 80 °C for 2 min at a rate of 30 °C, increased to 150 °C at a rate of 5 °C/min, then increased to 280 °C at a rate of 3 °C/min, and finally increased to 320 °C and held for 1 min"	[175]
Sediment		0.001–0.004	0.004–0.079			GC-MS/MS "Oven temperature: 80 °C, 2 min-1, 150 °C/min to 290, 0 min, 310 °C, final hold of 1 min. RT: 11.21–22.35 min"	[176]
Soil, Sediment	20	MDLs: Soil: 0.0020–0.058; sediment: 0.0013 to 0.50		Surrogate spike: 86-100	<10	GC-MS: "The GC temperature program was initially maintained at 80 °C for 2 min, and then increased to 180 °C at a rate of 20 °C/	[96]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						min, and then increased to 220 °C at a rate of 2 °C/min, and then increased to 245 °C at a rate of 1 °C/min, and then increased to 310 °C at a rate of 10 °C/min, finally held for 10 min"	
Sediment		0.005–0.048	0.018–0.159			GC Ultra TSQ Quantum XLS: "Oven temperature: 90 °C, 1 min,15 °C/min to 160 °C 3 °C/min to 225 °C, 6 °C/min to 305, 8 min, 3 °C/min to 310 °C, final hold of 17 min"	[177]
Sediment	15	0.0029–0.35		80-120; surrogate: 79 and 73	3.4–14	GC-MS: EIM mode, "initial at 80 °C hold for 2 min, 20 °C/min to 180 °C, 2 °C/min to 220 °C, 1 °C min $^{-1}$ to 245 °C, 10 °C min $^{-1}$ to 300 °C, hold for 10 min. RT: 17.569–29.913 min"	[97]
Sediment	13					LC-HRMS: "The full MS scan ranged from 100 to 900 *m/z*, and the collision gas was N$_2$. The capillary temperature and probe heater temperature were set at 325 °C and 350 °C, respectively, and the spray voltage was 3000 V, RT: 0.49–9.90"	[68]
Sediment	19	0.01–0.59		71–106	3–19	GC-MS: "The temperature program was: initial temperature: 60 °C, 6 °C/min to 290 °C (10 min). The carrier gas was nitrogen at a flow rate of 1.2 mL/min"	[178]
Sediment	18	0.04–0.20		Blank and sample spiked surrogate: 90.25–110.49	<35	GC-MS: "The oven temperature programming was set to start at 50 °C, holding for 3 min, increased to 300 °C at a heating rate of 12 °C/min, and holding to 45min"	[179]
Sediment	18	0.01–0.20		>87		GC-ECD: "initial temperature: 120 °C, 15 °C/min to 210 °C (1 min), 2 °C/min to 245 °C (1 min), 30 °C/min to 290 °C (3 min)"	[37]
Sediment	HCHs and DDTs	0.00188 to 0.013 for HCHs, 0.00017 to 0.0123 for DDTs		Surrogates: 51-85		GC-MS-ECNI	[180]
Sediment	7	0.02–0.16		Surrogate: 52–118; individual recoveries: 71–107; spiked sediment and blank: 75-112		GC-ECD: "The following oven heating ramp was used: (1) 100 °C for 1 min, (2) 100–140 °C at 5 °C/min for 1 min, (3) 140–250 °C at 1.5 °C/min for 1 min, and (4) 250–300 °C at 10 °C/min for 10 min (total run = 90 min)"	[102]
Sediment	22	MDL: 0.10–1.59	0.34–5.30	87–118		GC-HRMS: "120 °C(0.5 min) to 180 °C at 10 °C min. −1 to 210 °C at 4 °C min. −1 (12 min) to 300 °C at 10 °C min $^{-1}$"	[181]
Sediment	24	MDL: 0.004–0.13		Surrogate: 69.1 and 90.4		GC-ECD: "initial temperature was 100 °C for 1min, raised to 200 °C at a rate of 4 °C/min, then increased to 230 °C at 2 °C/min,	[182]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Sediment	Insecticides(34)	0.013–0.33		Spiked samples: 40–130; surrogate: 65.3–61.5		finally raised to 280 °C at 8 °C/min and held for 15 min" GC-MS: "The oven had an initial temperature of 70 °C holding for 1 min, and being heated to (i) 180 °C at 20 °C/min, (ii) 260 °C at 4 °C/min, and (iii) 300 °C at 15 °C/min, which was held for 6 min"	[103]
Sediment	19	MDL: 0.01–0.16		Surrogate: 93 ± 13 and 83 ± 7; spiked standard: 75–95	5–12	"The oven temperature was initiated at 100 °C (held for 1 min) and increased to 200 °C at 4 °C/min, 230 °C at 2 °C/min, and finally 280 °C at 8 °C/min (held for 15 min)"	[183]
Sediment	24	0.01–0.02		71.2–106		GC-ECD	[184]
sediment	17	0.001–0.005		Matrix: 70–109; procedural: 77-114		GC-ECD: "oven-heated started at 80 °C with a 2 min hold, ramped to 190 °C at a rate of 25 °C/min, then 5 °C/min to 280 °C, and finally 25 °C/min to 300 °C with a 5 min hold"	[185]
sediment	22	MDL:2.78 × 10^{-4}-0.020579		62–124	<20	GC-MS/MS	[186]
sediment	6			Spiked: 74.5–106.2; sur: 68.95		GC-MS/MS: "an initial temperature of 80 °C was maintained for 2 min and increased at a rate of 20 °C/min to 180 °C and held for 5 min. Then, it was increased at a rate of 10 °C/min to 290 °C and maintained for 15 min"	[187]
Sediment		0.00015–0.0003 ng/mL		72–110		GC-MS: "The oven temperature was programmed as follows: maintain 80 °C for 1 min, then increase at 20 °C/min to 150 °C and then at 5 °C/min to 300 °C, and then hold at the final temperature for 5 min"	[188]
Sediment	14	MS/NCI: 0.03–2.24; ECD: 1.2–4.6	MCI: 0.11–7.41L; ECD: 3.9–15.2	NCI: 88.6–120 %; ECD: 81.8–117 %	NCI: 0.2–1.7; ECD: 0.4–2.7	ECD: "Initial temperature 90 °C held for 1 min; ramped at 30 °C/min to 200 °C, not held, 2 °C/min to 235 °C, not held, 20 °C/min to 300 °C, held for 10 min, RT: 50–60 min"; GC-MS/NCI: "Initial temperature 40 °C held for 1min; ramped 50 °C/min to 110 °C, not held; 5 °C/min to303 °C, not held: 20 °C/min to 335 °C, held for 5.4 min. RT: 45–48 min"	[189]
Sediment	18	MDL: 0.056–0.918		Sur: 60-13	<20	GC-ECD: "Initial column temperature started at 100 °C, was kept for 1 min and raised to 320 °C at a rate of 4 °C/min and held for 5 min"	[190]
sediment	21	0.003–0.080		Spiked recovery: 81.3–94.2; surrogate recovery: 73.6–112.7 % and; 68.7–101.8	<15	GC-μECD and GC–MS: "Splitless, oven temperatures were programmed from 60 to 170 °C (at the rate of 25 °C/min, hold 2 min), from 170 to 250 °C (at the rate of	[86]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						3 °C/min, hold 4 min), from 250 to 280 °C (at the rate of 4 °C/min, hold 1 min), and finally from 280 to 300 °C (at the rate of 20 °C/min)	
Sediment	11	0.05		80.26–95.89		GC-MS/MS	[191]
Sediment	20	0.003–0.08		78–110	0.19–1.93	GC-μECD: "Splitless, the oven temperature programs for OCPs and PCBs were set as follows: holding at 60 °C for 1 min, heating to 170 °C at a rate of 10 °C/min holding for 2 min, and then heating to 280 °C at a rate of 5 °C/min holding for 2 min, and finally heating to 300 °C at the rate of 20 °C/min"	[192]
Sediment	25		MQL: 0.0025	53–119		GC-ECD: "The oven temperature was programmed to 50 °C for 1 min, increased at 20 °C/min to 200 °C and at 10 °C/min to 300 °C, and held for 5 min"	[193]
Sediment	HCH, DDT	MDL: 0.005–0.01		79 and 97		GC-ECD: "The oven temperature began at 60 °C for 1 min and increased to 290 °C (10 min hold time) at a rate of 4 °C/min"	[194]
Sediment	21	MDL: 0.003–0.86		78–110		GC-ECD: "The oven temperature was programmed from 60 to 170 °C (2 min hold) at the rate of 10 °C/min, to 280 °C (3 min hold) at the rate of 5 °C/min, and finally to 300 °C at the rate of 15 °C/min"	[195]
Sediment	13	0.25		75–115		GC-ECD	[196]
Sediment		MDL: 0.020–0.04		Matrix-spiked: 75–110; surrogate: 70-105		GC-MS	[197]
sediment	DDT		QL: 0,002	Sur: 55–92; spiked bl: 63–110; sample std: 76-101		GC-MS/MS: "Oven temperature ramps were programmed as follows: 50 °C for 1 min, increased at 20 °C/min to 200 °C, then at 10 °C/min to 300 °C, with a final hold of 5 min"	[198]
Sediment, worm	10		0.01–0.5	84–94		GC-MS: "The oven temperature programme was set at 90 °C where it was held for 2 min, it was ramped to 280 °C at a rate of 6 °C/min"	[199]
Sediment	21	0.003–0.080		Spiked recover: 81.3–94.2; surrogate recovery: 73.6–112.7 and 68.7–101.8	<15	GC-μECD: "Oven temperatures were programmed from 60 to 170 °C (at the rate of 25 °C/min, hold 2 min), from 170 to 250 °C (at the rate of 3 °C/min, hold 4 min), from 250 to 280 °C (at the rate of 4 °C/min, hold 1 min), and finally from 280 to 300 °C (at the rate of 20 °C/min)""	[86]
Sediment	22	MDL: 0.002–15.21		61 ± 8	0.4–20	HRGC/LRMS: "Split, oven temp: 75 °C (holding time of 1 min) to 100 °C at 25 °C min−1, then to 225 °C at 6 °C min−1 (holding time of 1 min), finally to 255 °C at 6 °C min−1 (holding time of 1 min); 30 min run time"	[200]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Sediment	22	0.04–0.71		82–94		GC-MS: "The GC oven temperature was programed as follows: initial temperature 70 °C (2 min), then raised to 175 °C (10 °C/min) and held isothermally for 4 min, then raised to 320 °C (5 °C/min) and finally held for 1 min. The injector was set from 50 °C (0.1 min isothermal) to 250 °C (200 °C/min) held for 10 min"	[201]
Sediment	20	0.03–0.2		Surrogate: 88.6 and 91.7 %		"Splitless, the initial temperature of the oven was 90 °C with a 1-min grip, and the temperature was increased to 210 °C at the rate of 10 °C/min with a 1-min hold; the temperature was enhanced to 230 °C with increments of 1 °C/min with a 10-min hold; ultimately, the temperature was increased to 250 °C at the rate of 1 °C/min"	[202]
Sediment, soil	4)	1.6 to 2.3; sediment: 0.2–4		Surrogate: 70-92		GC-MS: "Splitless, the oven temperature was programmed to 140 °C with a 0.5 min hold, ramping at 20 °C/min to 270 °C and a 5 min hold, then to 290 °C at 3 °C/min and a 2 min hold, and finally to 310 °C at 40 °C/min and a 3 min hold"	[203]
Sediment, soil	4	2×10^{-5}-5x10^{-5}		82–106			[204]
Sediment, soil	18	0.05–0.25		Spiked: 88-1-102.1	<6	GC-MS: "The column oven temperature was initially 50 °C for 1 min, ramped up at 30 °C/min to 150 °C for 1 min, 10 °C/min to 180 °C for 3 min, 3 °C/min to 210 °C for 1 min, 5 °C/min to 250 °C, and finally 15 °C/min to 290 °C for 3 min (RT: 36 min)"	[205]
Sediment	17	DL: 0.42–3.1		CRM: 85.1–123.4	spiked revovery: 8.9–22.9	GC X GC-TOFMS: "The GC oven was set to 75 °C (held for 0.5 min), then raised to 280 °C at a rate of 8 °C/min and maintained at this temperature for 2 min"	[206]
Sediment	13			Sur: 75-110		GC-MSD: "Oven temperature was set at 60 °C for 1 min and increased to 290 °C (10 min hold time) at a rate of 4 °C/min"	[207]
Sewage sludge	17	0.04–0.49	0.22–2.17	85.2–99.6	3.5–9.6	GC/μ-ECD: "The oven was held at 100 °C for 1 min, and ramped up at 20 °C per min to 180 °C, at 5 °C/min to 270 °C, and at 20 °C/min to 320 °C"	[208]
Sediment, plant	16	MDL: soil: 0.007–0.028; plant: 0.030–0.137		75-125; surrogate: 84.1–110.6		GC-ECD, GC-MS: "The temperature programming of oven is: 110 °C (hold 2 min), raising 10 °C/minutes to 180 °C, then 5 °C/minutes to 280 °C (hold 20 min)"	[209]
Sediment, shrimp	6	0.1–0.5		64.4–94.6	<10	GC-MS/MS: "The oven temperature was programmed to an initial temperature of 50 °C, held for 1 min, increased at a rate of	[210]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Water, Sediment	20	100–1500		65–110	<20	25 °C min^{-1} to 125 °C, and finally increased at a rate of 10 °C min^{-1} to 310 °C and held for 2 min" GC-MS: "Splitless, the oven temperature was programmed from 35 °C, increased to 150 °C with a ramping rate of 15 °C/min and held for 5 min, and then increased to 290 °C with a ramping rate of 3 °C/min and held for 2 min"	[211]
Water, sediment	8	0.07–0.31	0.21–0.95	92.4		GC-MS: "Splitless, the oven temperature program was: 70 °C raised to 140 °C (25 °C/min), a second ramp at 15 °C/min to 210 °C and a final ramp at 10 °C/min to 300 °C"	[212]
Water, sediment	19		Water: 0.8–2; sediment: 0.075–0.122	water: 71–106; sed: 77-110	<15	GC-ECD: "80–196 °C (4 °C/min rate, 2 min), from 196 to 224 °C (4 °C/min rate, 2 min), from 224 to 240 °C (4 °C/min rate, 2 min), and from 240 to 275 °C (rate 4 °C/min, 2 min)"	[213]
Sediment, SPM	7			70 and 106 %		"The GC temperature program started at 80 °C (1.5 min hold), ramped 40 °C/min to 200 °C (18 min hold), and finally ramped 5 °C/min to 305 °C"	[214]
DP SPM SED	16	D & SPM: 78.8–102.7; SED = 0.0005–0.0050		Procedural blank DP:80.5; SPM: 79.3; sediment 83.7. Spiked blank: 78.8–102.7			[215]
sediment, mud	9	0.5	<5	80–120	<35	GC-QqQ/MS: "the GC oven is temperature-programmed from 80 °C (isotherm 2 min) to 300 °C at 12 °C/min, then held for 5 min until run time 25.33 min"	[216]
Sediment, aquatic food	19	Sediment: 0.04–0.09; 0.03–0.21		Sediment: 81.7–109.8; aquatic food: 86.4–110.4		GC-MS: "column temperature was initially set at 80 °C for 1 min, ramped at 30 °C per minute to 170 °C, 5 °C per minute to 240 °C, 30 °C per minute to 320 °C, and then held at 320 °C for 3 min; RT: 10.55–17.96"	[217]
Soil air, and deposited sample	24	For PUF: 0.05, DP: 0.00005, Soil: 0.005		PUF:82; Filter: 79, DP: 100; Soil		GC-HRMS: "Splitless, the oven was held for 2 min, then increased to 160 °C at 10 °C/min, to 225 °C at 4 °C/min, and finally to 320 °C at 10 °C/min and held for 2 min"	[218]
Air, soil	10	MDL: soil: 7 × 10^{-5}–1.13 × 10^{-3}; air: 1 × 10^{-6}–4.6 × 10^{-5}		Surrogate: soil:70.2, 99.5; air: 75.4, 98.6	<10	GC-MS/MS: "The GC oven temperature was programmed as: initially 100 °C, 10 °C/min to 200 °C, 1 °C/min to 230 °C for 1 min, and 10 °C/min to 290 °C for 10 min"	[219]
Soil and water	8	10.4 (water); 490 (soil)	58.3 (water) 1.63(soil)	79.4–129	4.52–18.8	LC-QqQ-MS/MS; GC-MS/MS: "The column temperature and ion source temperature	[220]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m³)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m³)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						were controlled at 30 °C and 200 °C, respectively. N2 was used as nebulizer with 40 psi, 450 °C, and 11 L min⁻¹. The capillary voltage was controlled at 4000 V″	
Air	22	MDL: 0.000037–0.000269	0.118–0.940	61.2–114.5: CRM: 58.1–116.1		GC-HRMS: "The initial oven temperature was maintained at 100 °C for 1 min, which was then increased to 180 °C, 200 °C, 220 °C, and 300 °C at a rate of 20 °C·min⁻¹, 1.4 °C·min⁻¹, 5 °C·min⁻¹ (held for 0.314 min), and 40 °C·min⁻¹ (held for 4.4 min), respectively"	[221]
Air	1	0.01–0.05		79%-105		GC-MS-ECD: "The oven temperature program was as follows: the temperature started at 100 °C, held for 1 min, and then increased at 4 °C/min to 200 °C, at 2 °C/min to 230 °C, and at last at 8 °C/min to 280 °C, with a final holding time of 15 min"	[222]
Air	13	0.000011–0.000073	0.006–0.030	Surrogate:62-93		GC-MS: "The temperature program for GC started at 80 °C (1.5 min hold), then continued with 40 °C/min to 200 °C (18 min hold) and lastly 5 °C/min to 305 °C (no hold)"	[30]
Air	18	DL: 0.05		101	22	GC-HRMS: "The oven was held for 2 min, then increased to 160 °C at 10 °C/min, to 225 °C at 4 °C/min, and finally to 320 °C at 10 °C/min and held for 2 min"	[223]
Air	OCPs(27); CUPs (25)			OCPs: 38.2–148; CUPs: 59.3–170		GC-APCI-MS/MS: splitless, "the oven temperature programme was 90 °C (1 min hold), then 40 °C.min⁻¹ until 200 °C, followed by 2 °C.min⁻¹ until 240 °C, and 40 °C.min⁻¹ until 310 °C (5 min hold)"; GC-MS: "The temperature program for GC started at 80 °C (1.5 min hold), then 40 °C.min⁻¹ to 200 °C and finally 5 °C.min⁻¹ to 305 °C (no hold)"; **CUPs:** HPLC-MS/MS	[51]
Air	4	IDLs: 0.011–0.071 pg[a]; MDL: 0.000005–0.00111		Ave:108	<15	GC-MS/MS	[224]
Air	10	0.24–4.83 pg[a]		>70		GC-ECD	[225]
Air	25	0.000002–0.0007		Surrogate: 33.9–155	0.64–16	GC-HRMS: "The oven temperature was 110 (1 min), 20 °C/min up to 210 °C , 1.5 °C/min up to 218 °C (1 min), 2/min up to 260 °C (1 min)"	[226]
Air	29			Samples: 3–210; method blank: 11–135; fields: 12-141	0–17	GC-MS: "The programmed oven temperature went from 55 °C (hold time 2 min) to 200 °C at 70 °C min⁻¹ (hold time 1 min), and then to 280 °C (10 °C/min) (hold time 1 min)., and then to 310 °C at 10 °C/	[227]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Air	33	0.02–0.3		82–106	2–9	min and finally to 325 °C at 70 °C (hold time 10 min)" GC-MS/MS: "The programmed oven temperature went from 90 °C (hold time 5 min) to 180 °C for 5 min, and then to 180 °C (5 °C/min) (hold time 3 min)., and then to 280 °C at 10 °C/min and finally to 300 °C (hold time 3 min)"	[228]
Air	17	0.0001–0.055		Surrogate: 118-123		GC/ECNI-MS	[229]
Air	OCPs(27), CUP (3)	2×10^{-5}-3x10^{-4}	5×10^{-5}-1.03 $\times 10^{-3}$	Surrogate: 75 and 105		GC-ECD	[230]
Air	9	0.71–2.13; IDL: 0.04–0.15		69–117		GC-μ-ECD: "The initial oven temperature was held at 50 °C for 1 min and raised to 200 °C at 25 1 C/min, 200–300 1C at 8 °C/min"	[231]
Air	13	MDL: 0.007–1.27 ng/sample[a]		Int stan: 44.1–211		GC-MS: "The GC temperature program started at 80 °C (1.5 min hold), then continued with 40 °C min1 to 200 °C (18 min hold) and lastly 5 min1 to 305 °C (no hold)"	[232]
Air	100	IDL: 0.04–4.83 pg[a]		>75		GC-ECD: "the oven program for OCPs was 80 °C (1 min) and then 20 °C/min to 300 °C. The injector and detector inlet temperatures for both PCBs and OCPs were 250 °C and 320 °C, respectively"	[233]
Air	15		MQL: 0.00080–0.097	40–110	<10	GC-MS: "The oven temperature program was as follows: 80 °C for 1 min, increased at 30 °C/min to 190 °C, followed by 2.5 °C/min to 230 °C, then increased at 20 °C/min to 260 °C and hold for 12 min, finally followed by 20 °C/min to 320 °C and hold for 23 min"	[234]
Air		0.21		78.2–93.1	7.48–17.1	GC × GC-LRMS ECD: "The initial oven temperature was maintained at 100 °C for 1 min and then 30 °C/min to 160 °C maintained for 5 min and increased to 300 °C at 1.5 °C/min and maintained for 2 min"	[235]
Air	20			81.4–115.31		GC-ECD: "The column temperature was increased from 80 °C to 210 °C at 10 °C/min, then increased at 0.8 °C/min to 250 °C and held for 1 min, and finally increased to 290 °C at a rate of 10 °C/min and held for 12 min"	[236]
Air suspended particles	16	MDL: 0.0005–0.0027		66–108		GC-MS: "The oven was operated with a temperature ramp: 40 °C for 1 min, 50 °C/min up to 110 °C, 5 °C/min up to 303 °C, 20 °C/min up to 335 °C for 20 min"	[237]

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Air, water	4	DP: 0.1–5.94; GP: 1.8–5.52	DP: 0.11–7.15; GP: 2.31–8.31			GC-ECD: "The programmed oven temperature went from 90 °C (hold time 1 min) at 190 °C at 20 °C min^{-1}, and then to 310 °C (3 °C/min) (hold time 18 min)"	[100]
Air, water	12	Air: 1×10^{-7}-1.4×10^{-6}; Water: 1×10^{-7}-$7x10^{-7}$		Surrogate: 45–64 and 72–98		GC-MS/MS: "Splitless, the GC temperature programme was 80 °C (1 min hold), then 40 °C min-1 to 200 °C, and finally 5 °C min-1 to 305 °C".	[104]
Air, water	9	IDL: 7.7×10^{-5}-9.6×10^{-5}; MDL: Air: 0.01342–0.0175; water: 1.991×10^{-5}-2.649×10^{-5}				GC-ECD, GC-MS: "Temperature program: 60C, 1.5 min, 25C min1, 140C, 8C min1, 300C, 20 min"	[238]
Air, seawater, coral tissues	22	MDL: Seawater: 2.8×10^{-9}-5.64×10^{-8}, Air: 9×10^{-7}-1.85×10^{-5}, Coral tissue: 0.001–0.0358		Surrogate: 63.2–110		GC-MS/MS	[239]
Air, soil	22	IDL: 2.5×10^{-5}-5.31×10^{-4}		64.5–95.0; CRM: 73.1–152		GC-MS: "The GC oven temperature program was as follows: 50 °C hold for 1 min; 25 °C/min to 100 °C; 5 °C/min to 260 °C (hold 1 min); 10 °C/min to 300 °C (hold 5 min)"	[240]
Gas-phase, particle phase	22	0.00112–0.0076	0.00232–0.0054	64–103		GC-MS: "Splitless, temperature program was 2 min at 80 °C, 10 °C/min to 285 °C (wait for 5 min), 25 °C/min to 315 °C (wait for 5 min)"	[241]
Particle-bound, sea	20	MDL: 1.5×10^{-6}-3.3×10^{-4}		76–81		GC-MS: "The oven temperature was programmed as follow: 80 °C at the beginning and held for 2 min, increased to 180 °C at a rate of 20 °C per min, increased to 220 °C at a rate of 2 °C per min, increased to 245 °C at a rate of 1 °C per min, increased to 310 °C at a rate of 10 °C per min and held for 10 min"	[242]
Gas, seawater	221	Gaseous: 2×10^{-6}–0.038; aqueous: 3×10^{-9}–7.6×10^{-5}		Gaseous: 44–128; seawater: 42–127; median recoveries in spiked samples: 52–110 in air and 43–136 seawater		GC-MS/MS: "Splitless, the GC column temperature was initiated at 60 °C (held for 1 min), increased to 120 °C at 40 °C/min (held for 0 min), then to 310 °C at 5 °C/min (held for 0 min). RT: 5.6–56.5 min"	[243]
Gas/particular matter phase	31	MDL: 1–345 fg/m^{3a}		surrogate 81–121	6.5–15.1	GC-MSD	[244]
Gas phase	17	MDLs: 0.0007		87–101.8	<5	GC-ECD	[245]
Gas-particule	23	MDL: 1.1–344.4 fg/m^{3a}		83–120	7.4–13.7	GC-ECNI: "oven program was set to 50 °C initial temperature, hold for 1 min, 30 °C/min to 140 °C, then 2.20 °C/min to 285 °C, and finally 15 °C/min to 300 °C, hold for 15 min (85.91 min total run time)"	[246]
Aerosol		0.14–0.44	0.63–1.5	90–14	0.19–3.4	GC-MS	[247]
Silicone sheet	7	0.036–0.173	0.0008–0.078			GC-MS/MS	[248]

(*continued on next page*)

Table 2 (*continued*)

MATRIX	No. of COMPOUNDS	LOD (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	LOQ (liquid: ng/mL; Solid: ng/g; Air: ng/m^3)	RECOVERY(%)	RSD (%)	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
plastics	18	30–500		Surrogate: 74.4–80		GC-MS: "The GC oven temperature was raised from 70 °C (held for 2 min) to 200 °C at 20 °C/min and then increased to 250 °C at 10 °C/min"	[249]
plastics	8			Surrogate: 82.5–114; spike: 95-105		GC-ECD: "The column oven temperature was programmed as follows: 80 °C for 2 min, increased at 8 °C per minute to a final 280 °C, which was held for 20 min. Total run time was 49 min"	[250]
Water, microplastics	8	0.2–10		69–85		GC-MS, GC-HRMS: "The oven temperature was initially set at 75 °C for 2 min, increased at first to 150 °C at a rate of 20 °C/min and held for 2 min, then increased to 260 °C at a rate of 3 °C/min and held for 2 min, and finally increased to 300 °C at a rate of 20 °C/min and held for 1 min"	[251]

[a] = values not converted.

3.2. Influence of properties of OCPs on analysis

The environmental fate of OCPs is determined by their physical-chemical characteristics and its analysis can be greatly influenced by the features of OCPs [9]. The analysis of these POPs therefore requires a thorough understanding of the partition coefficient, volatility, solubility, and potential initial interferences of OCPs. It can assist researchers and analysts in streamlining their extraction and purification processes, producing more accurate and reliable results. Water, sediment, and biota may all be distributed differently depending on the partition coefficients and hydrophobicity [61]. Pesticides are persistent in the environment due to their solubility, mobility, degradation half-life (DT_{50}), n-octanol-water partition coefficient (Kow), as well as the characteristics of the soil [9]. However, monitoring studies rarely address the correlation between the presence of pesticides and their properties [62]. The octanol-water (K_{OW}) partition coefficient is the ratio of a substance's equilibrium concentration in two immiscible phases [63], such as the concentration of OCPs in water and lipid-based compounds. The pesticide has a stronger affinity for lipid-based compounds than for the aqueous phase, according to a high partition coefficient [9]. Because of this, it is demanding to extract the pesticide from the sample matrix in the analysis of OCPs with high partition coefficient like sediment [64]. The parameters, such as the selection of cleanup sorbent and the quantity of cleanup sorbent, that affected the partition of analytes among the various matrices were optimized. Achieving satisfactory recoveries, better LOD performance, and high sensitivity in high-fat matrices (soybean, peanut, rapeseed, and sesame seeds) analysis are all possible with the methylamine modified graphene (CH_3NH-G) based dispersive solid-phase extraction (d-SPE) cleanup approach [65]. Organochlorine pesticides are semi volatile [66]; hence, It's crucial to reduce pesticide loss caused by volatility while analyzing OCPs. Controlling the temperature and pressure while the sample is being prepared and analyzed and using sealed sample containers can be utilized to stop OCPs from escaping during storage and transportation [67]. The choice of analytical techniques may be influenced by the volatility of some OCPs compared to others as would be described later. Another significant variable that might affect the analysis of OCPs is solubility. OCPs are known to be hydrophobic and lipophilic [4]. Therefore, high water content may retard the solubility thus influencing their rate of recovery. Since it affects both the rate and duration of compound volatilization in sediment, the water content of sediment has a significant impact on extraction [68]. Furthermore, a sample matrix can interfere in a variety of ways, including by co-eluting matrix compounds with the target OCPs, suppressing or enhancing the signal due to matrix compounds, and creating artifacts or breakdown products during sample preparation and analysis [69]. Many approaches can be applied to reduce the interference caused by the sample matrix and give good recover and lower detection limits. One strategy is to critically choose an appropriate extraction solvent as described previously.

3.3. Recovery

Recovery value is the measure of how well an analytical technique can determine the concentration of an analyte in a sample, and should always be established during method validation [70]. Poor recovery can result in erroneous results and compromise the accuracy and reliability of the analysis and can occasionally result in false negatives. Analyte breakdown in the instrument can result in low recoveries, for example DDT and endrin are most likely to breakdown in the GC inlet [71]. Large matrix effects have been reported by the US Department of Agriculture in residue analysis at low spike concentration (with 50–150 % recoveries) which cannot be entirely removed by cleanup procedures (Dana Ayu Mustofa et al., 2022). Unusual recoveries of 200.54 % from hair samples [72], 15–144 % from owl feathers [73], 5, 9, 36 % [74], 172.6, 169.3, 180.9, 140.7, and 167.3 %, which was thought to be mostly caused by the significant variances between the soils [75], and 69–140, 8.2–41.6 from soil [76], have been recorded. Several recovery techniques are reported (Table 1), such as procedural blank, spiked blank, spiked sample, and surrogate recovery among others. Surrogate standard is used to evaluate the loss of contaminant throughout the analysis process and determine the matrix effect [77]. The absence of OCPs in procedural and field blanks implies minimal contamination throughout transport, storage, and analysis [78]. Although a recovery factor that is as close to 100 % as possible is ideal, there is no set minimum. Therefore, provided the sensitivity of the method is appropriate, an analytical method with low recovery may be suitable for a particular analyte [79]. Extremely low recoveries were observed in snow (27 %) [80], glacier-ice core (9.67 %) [81], water (22.42 %) [82], and air (38.2 %) [51] (Table 2). Samples with poor recoveries are often discarded, as was the case with 220 samples that have <40 % recoveries in one study [76], likewise HCB with recovery 3.35 % and other recoveries <60 % were discarded [15].

3.4. Relative standard deviation (RSD)

The relative standard deviation (RSD) is a statistical measure commonly used in Analytical Chemistry to indicate the precision of a measurement. It aids in evaluating the consistency and reliability of the findings produced by a measurement method. It is calculated as the ratio of the standard deviation (SD) to the mean value of the same data set, expressed as a percentage. Replicate injections ranging from 3 to 10 have been used to determine the RSD (Table 1). However, minimum of 5 replicate injections is required [252]. A low RSD indicates high precision, while a high RSD indicates low precision.

3.5. LOD and LOQ

The LODs are measures of the lowest level of a pesticide that can be detected with acceptable precision and accuracy under specified test conditions [253]. Researchers have defined LODs in various ways, such as the logarithm of odds estimated on the basis of signal-to-noise ratio of 3 (3(S/N)) [205], the lowest concentration that produced a chromatographic peak that was three times larger than the background noise [48], higher value between the IDL and LOQ of blank samples [254], lowest addition level for the recovery

analysis [85], amount of analytes per sample corresponding to the lowest calibration [255,256], and the concentration of analytes in a sample that yielded peak S/N [19,257]. The LOQ on the other hand is the lowest amount of a particular type of pesticide in the tested sample that can be determined under the specified test conditions with acceptable precision and accuracy [258]. While LOD is useful for figuring out the minimal detectable concentration of an analyte, it does not reveal information about the precision and accuracy of quantification at low concentrations. However, LOQ is a more helpful measure for technique validation and data analysis because it offers information on both sensitivity and accuracy [259]. Regarding the term most appropriate to define this measure, there has

Table 3
Limit of detection (LOD) and limit of quantification (LOQ) calculations.

S/N	LOD	MDL	LOQ	Reference
1	3σ		10σ	[17]
2	3SD			[255,122, 274–276]. [98, 256]
4	concentration of native Component in the quantification standard divided by 3S/N			[26,87],
4	3SD		10SD	[189]
5	mean of solvent blank + 25 ppb of surrogate standard, + 3SD			[275]
6			10S/N	[85]
7	2SD		10SD	[277]
8	S + 3.3σ		3*LOD	[93]
9		Sum of average of blank data and 3SD	10SD (i.e. MQL)	[177]
10		Average of all blanks + 3SD		[73,94–97,278]
11	Average lab blank +3SD			[51,66,145,229, 279]
12		SD of the replicate analysis * one-tailed t statistics for the 99 % confidence interval		[203]
12	Mean of 10 blank + 3 fold of their SD		Mean of 10 blank + 10 fold of their SD	[280]
13	Lowest calibration point of an amount producing a S/N of 3.3		Lowest calibration point of an amount producing a S/N of 10	[30]
14	Lowest calibration standard with the S/N > 3		Lowest calibration standard with the S/N > 10	[74]
15	3 N/S		10 N/S	[19,52,88,257, 149,281]
16		Student's t-value of 3.14 * SD of spiked blank		[91,282]
17		3SD	10SD	[181]
19		Mean blank+3SD or 1/2IDL		[283]
20	IDL calculated from linear extrapolation based on the lowest calibration standard	Average of field and method blank concentration+3SD		[232,240,241, 284],
21	Average amount of each analyte in blank +5SD+3SD			[100]
22	3*baseline noise in the chromatogram		5*baseline noise in the chromatogram	[29] Or [285]
23	SD of the blank matrix samples with the lowest concentrations of target standards by 7 replicates			[103]
24		3SD blank conc		[104]
25	3σ_b/S			[36]
26	3.3 SD of the smallest amount of the analyte that gave a S/N ≥ 3			[28]
27	3σ_b/b		10σ_b/b	[286]
28			Mean + student t-test (3.75) SD of field blank sample	[254]
		IDL = 3S/N; MDL = IDL/sample's average mass		[89,239]
29			9S/N	[27]
	3S/N		9S/N	[213]
			highest detected value	[287]
30	3.3(S/b)			[288]
31	3.3σ/S		10σ/S	[267,289],
32	Sy/S	(LOD*final volume)/(sample mass*injected volume)		[190]
33	Student's t-value of 3.14 * SD of spiked blank			[26,90],

σ_b = standard deviation of the intercept/average normalized intensity of the blank samples or; S = slope; b = slope of calibration curve; SD = standard deviation; LOD = limit of detection; LOQ = limit of quantification, MDL = method detection limit; MQL = method quantification limit; DL = detection limit; Sy = Standard deviation of the instrument for each analyte calculated using STEYX; S = slope of the linear regression of the calibration curve.

Table 4
Quality control/assurance in biological samples.

MATRIX	No. OF COMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/ INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Vegetables	11	0.07–0.18		76.21–93.51	4.76–11.1	GC-ECD: "Splitless, the initial temperature at 100 °C for 1 min, ramping to 190 °C at 12 °C/min, and held for 8 min, followed by continuous ramping to 250 °C at 3 °C/min, and held for 10 min"	[290]
Vegetable	10	0.0011 to 0.021	0.0013–0.034	95–110		HPLC: "40 % A at 70.1–75.0 min and 7 % A at 75.1–90.1 min. The column temperature was maintained at 30 °C"	[291]
Vegetables	20	0.01–0.08	**0.03–0.24**	88.6–102	<6	GC-MSD: "Splitless, Initial oven temperature was 150 °C, increase to 280 °C at 6 °C/min and final temperature was 300 °C"	[292]
Leafy vegetable (spinach, lettuce, oilseed rape, cabbage)	8	0.15–0.32	0.45–0.96	78.6–107.7	1.1–7.5	GC-MS: "Splitless, the oven temperature program was set as follows: 50 °C as initial temperature, maintained for 1 min, raised to 200 °C at 20 °C/min, raised to 230 °C at 5 °C/min, and maintained for 5 min, raised to 280 °C at 10 °C/min, and held for 1 min. The total run time was 25.5 min"	[293]
Vegetable	15	20–4500		60–120	0.2–19.8	GC-ECD: "Splitless, the oven temperature was kept at 90 °C for 1.0 min and then programmed at 3.5 °C min-1 to 170 °C followed by a final ramp to 280 °C at 5.0 °C min-1"	[294]
Vegetable oils	20	0.10–1.84		44–159		GC-MS/MS: "Splitless, oven temperature: 50 °C for 1 min, increase at a rate of 25 °C/min up to 125 °C, then increase at a rate of 10 °C/min up to 300 °C and held for 2 min"	[295]
Vegetables and fruits	19	0.019 to 0.033 (vegetable) 0.017–0.038 (fruit)	0.048–0.081 (fruit) 0.049–0.088 (vegetable)	87.2–99.27 (vegetables); 89.54–100.3 (fruits)		GC-MS/MS: "The temperature profile for GC was ranged from 80 °C to 290 °C. RT 14.23 30.54"	[48]
Fruits and vegetables	11	12–987	40–3290	79.72–104.83	0.48–11.58	"The oven temperature was programmed initially at 60 °C for 1 min, then raised to 140 °C	[270]

(*continued on next page*)

Table 4 (*continued*)

MATRIX	No. OF COMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/ INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Vegetables and fruits	19			91.31–99.29		at 12 °C min−1, and finally raised to 280 °C at 8 °C min−1" GC-PFPD: "Split, The oven temperature was maintained initially at 60 °C for 2 min, first Ramp at 10 °C/min to 200 °C, then second Ramp at 8 °C/min to 300 °C and held constant at 5 min"	[88]
Root of ginseng	20	0.18–2.0	0.55–6	51–156	1.8–12.6	GC-μ-ECD, GC-EI-MS/ MS	[296]
Raw and dregs Ginseng	5	Liquid: 100–400; soild: 0.0003–0.0012	Liquid:20–120; soild:0.001–0.004	Liquid: 70.3–85.6; solid: 83.4–106.9	Liquid: 2.7–6; soild: 4.7–14.9	GC-MS/MS: "The column temperature was 40 °C at the start, held for 1 min; increased to 120 °C at a rate of 30 °C/min; increased to 180 °C at 20 °C/min; and increased to 280 °C at 15 °C/min, maintained for 10 min; GC-ECD: The column temperature was 120 °C at the start, held for 1 min; increased to 150 °C at a rate of 8 °C/min, held for 2 min; and then increased to 270 °C at 4 °C/min, maintained for 7 min. RT: 4.83–7.34 min""	[297]
Fruits and vegetables	6	0.03–0.30	0.11.0	73.7–111.6	3.4–11.9	GC-ECD: "Splitless, initial at 70 °C, then heating at 5 °C/min to 135 °C, at 2 °C/min to 140 °C (held for 1 min), and finally ramped to 280 °C at 30 °C/min. Ultrapure nitrogen (99.999 %) was used as the carrier gas at 1.2 Ml min−1"	[281]
Fruits, vegetables	20	10–100	100–10 × 10^4	44–101		GC-ECD: "oven temperature 80 °C for 2 min ramped to 180 °C @ 20 °C/min up to 230 °C @ 5 °C/ min and finally up to 280 °C @ 20 °C/min and held for 5 min"	[69]
apple, peach, Chinese cabbage and cucumber samples	11	0.01–0.20	0.03–0.67	83.7–124	2.1–10.9	GC-ECD: "The initial oven temperature was set at 90 °C, followed by increasing it to 184 °C at 30 °C/min, to 198 °C at 1 °C/min and to 280 °C at 20 °C/min"	[84]
Foods, fruits and vegetable	19		0.009–0.156	24.3–154.7		GC–NCI–MS: "The temperature was immediately	[298]

Table 4 (*continued*)

MATRIX	No. OF COMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/ INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						increased to 240 °C at a rate of 3 °C/min, maintained for 5 min, then increased to 300 °C at 10 °C/min and maintained for 5 min"	
cereals, vegetables, and fruits,	20		0.009–0.156	31.5–92.3		GC–NCI–MS: "The temperature was immediately increased to 240 °C at a rate of 3 °C/min, maintained for 5 min, then increased to 300 °C at 10 °C/min and maintained for 5 min"	[299]
meat products, aquatic foods, dairy products, edible oils, chicken eggs, fruits, vegetables, and cereals	18)	0.0001–0.0004	0.0003–0.001	83–112	4–18	GC- Mecd: "The oven temperature was 75 °C (0.5 min) initially and increased to 300 °C at 10 °C/min (2 min) for a total run time of 25 min"	[300]
fruits, vegetables, cheese, yogurt, egg, meat, and fish, samples	13			7–95	0.6–4.4	GC-MS/MS "The GC temperature programme was 80 °C (1 min hold), then 15 °C per minute to 180 °C, and finally 5 °C per minute to 300 °C (5 min hold). RT 11.05–32.99 min"	[49]
Yam, cassava, cocoyam, sweet potato	14	0.45–3.51	3.94–10.69	86.56–98.74		GC-TOFMS: "Splitless, oven temperature from 70 °C (2 min hold), then raised to 130 °C at the rate of 25 °C/min, afterwards raised to 220 °C at 2 °C/min, and then raised to 280 °C at 10 °C/min, and eventually 4.6 min hold. RT: 5.58–17.5 min"	[301]
Carrot, Onion, Cabbage, Garlic and Ginger	17	0.017–0.405	0.17–4.05	86.64–93.86		GC-ECD "initial oven temperature of 150 °C which was later increased to 280 °C at 6 °C/min and the total run time was 21.67 min. RT: 5.117–18.875 min"	[302]
Water, fruits and vegetable	7	0.0005–0.004	0.0015–0.015	Water: 69.61–121.95; veg: 59.83–132.67; fruit: 67.41–115.37	Water: <9.76; veg: 10.37; fruit: <10.03	GC-ECD: "Split, The oven temperature program was started at 160 °C for 0.5 min, raised to 180 °C at a rate of 20 °C min–1 and held for 0.5 min, raised to 190 °C at 20 °C min–1 and held for 0.5 min, raised to	[303]

Table 4 (*continued*)

MATRIX	No. OF COMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/ INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						250 °C at 40 °C min–1 and held for 4 min, and then to 300 °C at 25 °C min–1 and held for 2 min"	
Tobacco leaves	20)	2–65.5	6.5 to 198	70–100	2.9–64.8	GC-ECD: "Splitless, the GC oven temperature was programmed as follows: 100C (0.5 min), 35C min1 to 220C, 10C min1 to 240C, 2C min1 to 250C, and 10C min1 to 290C (2 min). RT: 6.542–15.628"	[304]
Needle leaves	11	2.3×10^{-4}-0.026		52–117		HRGC-HRMS: "The oven temperature was 60 °C initially, held for 1.5 min and ramped at 10 °C/min to 140 °C, then increased to 220 °C at 4 °C/min, and ramped at 2 °C/min to 250 °C, and finally ramped to 300 °C at 8 °C/min"	[305]
Pine needle	10	0–3.25 pg[a]; IDL: 0.04 pg[a]	0–9.087 pg[a]	50–120		GC-MS: "initial temperature 50 °C (1min), 25 °C/min up to 200 °C, 8 °C/min up to 300 °C, 5.5 min stayed, 5 °C/min rise to 310 °C for 3 min"	[306]
Meat	19	0.27–1.51	1.10–5.20	81.6–116.3	1.78–18.2	GC-MS: "Split, Column 50 °C (0.4 min hold) to 195 °C at 25 °C/min; hold to 265 °C for 1.5 min at 8 °C/min; maintained at 315 °C for 1.25 min at 20 °C/min"	[307]
Walnut, soil	21	0.1–0.5	0.1–15.76	80–110		GC-MS/MS: "Splitless, the initial temperature of 70 °C was maintained for 2 min, and then ramped up to 150 °C at 25 °C/min, then to 200 °C at 3 °C/min, and finally to 250 °C at 8 °C/min for 10 min. RT: 9.19–29.41"	[85]
Rice, soil	22	0.02–0.76	0.06–2.6	Surrogate: 76-110		GC-ECD: "Oven temperature program was initially set at 80 °C and held for 2 min, then increased to 195 °C at the rate of 10 °C/min. The increased temperature ramped to 230 °C at the rate of 3 °C/min, finally ramped to 310 °C at the rate of	[308]

Table 4 (*continued*)

MATRIX	No. OF COMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/ INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						10 °C/min, and held for 5 min"	
Therapeutic tea	17	0.4–5.1	Spiked samples: 89.9–102.2		<12	GC-ECD: "Splitless, the initial oven temperature was 150 °C and was increased to 280 °C at 6 °C/min, RT 22 min"	[309]
Tea	20	0.16–2.06	0.54–3.84	80–94	2.43–2.93	GC-MS: "The column temperature programs s were as the following: 70 °C (2 min), 150 °C (25 °C/ min), 200 °C (3.0 °C/ min), 280 °C (9.0 °C/ min), isotherm (10 min), the total run time for the OCPs was 40.8. 70 °C (1.2 min) and the ramp rate was 10 °C/min to 280 °C (18 min)"	[310]
Tea	9	60–720	30–2400	75.87–111.56	0.8–9.0	GC-ECD: "Oven temperature program was: initial temperature of 80 °C, ramped at 30 °C/min to 180 °C, ramped at 3 °C/min to 205 °C, held for 4 min, ramped at 20 °C/min to 290 °C, held for 8 min, ramped at 50 °C/ min to 325 °C. The total GC run time was 27.92 min"	[269]
Tea	13	1.4–7.2		86.1–100.3	3.1–11	GC-MS: "The initial temperature was 50 °C, which was increased to 180 °C at 10 °C/min and maintained for 4 min. It was then increased to 210 °C at 2 °C/min and held for 4 min. Finally, it was raised to 231 °C at 3 °C/min and held for 5 min"	[311]
Corn, Corn floor, barn	26	MDL:C: 0.01–3.45; cf: 0.01–2.83; B: 0.01–1.98		48-129; 37–159; 35-135		GC-MS/MS: "The oven temperature was programmed as follows: the initial temperature (50 °C) was held for 1 min and increased by 25 °C/ min to 125 °C and then increased by 10 °C/min to 300 °C and held for 2 min"	[122]
Maize, flour	19	14.23–32.31	63.68–100.98	66.74–100.65		GC-MS: "The initial temperature was 90 °C for 2 min and later increased to 260 °C at 5 °C/min and held for 5 min"	[93]

Table 4 (*continued*)

MATRIX	No. OF COMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/ INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
maize	17	1–4	3–12	89.6–100.91	0.69–12	GC-ECD: "Oven temperature started at 150 °C and increased to 280 °C at 6 °C per minute. The injection was through a splitless injector, using helium as a carrier gas at a flow rate of 2 mL/min. The run time was 21.67 min"	[50]
Maize, sediment, water	23	Soil & maize: 0.00117–0.470; water: 2.4×10^{-7}–7.83×10^{-5}		Surrogate: 65.7–101		GC-MS/MS: "The GC oven temperature was set at 100 °C for 0.5 min, then 20 °C/min to 160 °C, 4 °C/min to 290 °C, and finally 10 °C/min to 300 °C, and hold for 10 min"	[266]
Water, plant and substrate	9	MDL: Water: 0.005 to 0.01; Plant: 0.01–0.05; substrate: 0.05–0.1		Spiked: water: 72.4–116.5; plant: 69.9–109.2; substrate: 72.4–116.5		GC-MS: "The column temperature was initiated at 80 °C (kept for 1 min), increased to 230 °C at 10 °C·min−1 (kept for 4 min)"	[312]
Water, plant, soil	35	Water: 0.0025–0.35; plant: 0.05–7.0; soil: 0.05–7.0				GC–MS/MS and UPLC-MS/MS: "Splitless, an initial temperature of 75 °C and a maximal temperature of 300 °C at the end of the injection's transfer phase (rate: 10 °C/s in 2.5 min)"	[313]
Plant tissue, soil	6	Plant: 25; soil: 100	Plant: 0.1			GC-ECD: "The temperature of the thermostat column was programmed from 40 °C (hold time 1 min) to 160 °C (hold time 3 min) at a heating rate of 20 °C/min, followed by heating to 250 °C (hold time 5 min) at 3 °C/min"	[314]
Plant	11		3.2–9.9	82.3–91.9	6–12	GC-ECD: "The initial column oven temperature was 150 °C; it was then ramped up at 2.5 °C per min to 270 °C and kept for 15min"	[315]
Plant	18	0.001–0.004	0.004–0.011	81–96	0.1–1.5	GC-qMS: "The oven temperature was programmed from 80 °C (initial time, 2 min) to 205 °C at a rate of 30 °C/min (hold time 5 min) and then heated to 290 °C at a rate of 10 °C/min with a final holding time of 3 min"	[316]

Table 4 (*continued*)

MATRIX	No. OF COMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/ INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Dietary supplement extract	8	0.00899–0.0931			4.48–12.9	GC-MS: "The GC oven temperature was ramped at 10 °C/min from 40 °C to 290 °C, and then held at 290 °C"	[317]
Kaht plant	15	DDT: 0.1; HCH: 0.15–0.45	HCH: 0.3; HCH: 0.5–1.5	86.72–114.75 (DDX),; 97.33–111.12 (HCH)	2.84–11.7	GC-MS: "The initial oven temperature was set at 90 °C and held for 3 min. The temperature was ramped to 150 °C at a rate of 15 °C/min. Then, it was ramped to 280 °C at a rate of 5 °C/min and held for 3 min"	[265]
Tree bark		3.85 × 10^{-5}–0.0131		51–98			[318]
Seaweed	20	0.001–0.004	0.005–0.017	72–120	<12	HPLC: "The column temperature was maintained at 35 °C"	[19]
seaweeds	17	1		80–108		GC-MS: "initial oven temperature at 90 °C for 0.5 min, which was then increased to 280 °C at 8 °C min-1 and again to 300 °C at 15 °C min-1 for 2.5 min. RT: 13.65–22.74 min"	[319]
wheat	15	0.02–0.03	0.010–0.05	80–92	2–8	GC-MS: "initial 30 °C (2 min hold), ramped to 180 °C at 15 °C/ min, ramped to 190 °C at 2 °C/min, ramped to 290 °C at 2.5 °C/ min, and ramped to 320 °C at 5 °C/min (15 min hold)"	[320]
Olive oil	10	0–3.25 Pg[a] IDL: 0.04 Pg[a]	0–9.087 Pg[a]	83–117		GC-MS: "The oven temperature analysis was as follows: the furnace was kept at 80 °C for 1 min and then, increased to 300 °C with 20 °C/ min"	[321]
Tap water, river water, palm oil mill effluent	Endosulfsn, dieldrin	0.0073, 0.0086	0.022, 0.025	98.6-1-3.5	4.61–6.79	GC-ECD: "The oven was temperature-programed from 120 °C to 190 °C at 40 °C/min, then to 285 °C at 30 °C/min"	[322]
Honey	2	1.0 and 2.0	2.0 and 4.0	84.1 and 111.7	1 and 8	GC-MS: "The GC oven temperature was programmed at 100 °C, with a heating rate of 20 °C/min to 200 °C (1 min) and with a heating rate of 10 °C/min to 280 °C (1 min), in a total analysis time of 15 min"	[323]

Table 4 (*continued*)

MATRIX	No. OF COMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/ INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Honey, agricultural soil	Pesticides (10)	Soil: 0.04–0.2; honey: 0.05–0.20		Soil: (64–90); honey: (73–99)		GC-ECD: "Initially, the column temperature was set at 90 °C for 1 min and then increased to 180 °C at 30 °C/min. It was then further raised to 260 °C at 4 °C/min and then kept at this temperature for 16 min"	[262]
Honeybee, pollen, honey	10				0.021–0.338.	GC-μECD: "initial furnace temperature is 80 °C (1 min), then with increases of 20 °C min-1 to 240 °C and 5 min wait at this temperature, then with increases of 5 °C min-1 to 270 °C and increases of 20 °C min-1 to 300 °C and kept at this temperature for 3.5 min"	[279]
Honey and propolis	13		Propolis: 0.49–1.11; Honey: 0.54–1.02	Propolis: 85.9–105.6; Honey: 89.9–106.3	Propolis: <14.4; Honey: <12.9	GC-MS: "Splitless, The GC oven was initially maintained for 2 min at 70 °C, and then the temperature was increased to 150 °C at 25 °C/min. Then, it was increased to 200 °C at 5 °C/min (held for 5 min), increased to 270 °C at 5 °C (held for 2 min), and finally increased to 290 °C at 25 °C/min (held for 5 min)"	[324]
Propolis	13	0.16–0.37	0.49–1.11	85.9–107.2	<11.5	GC-MS: "The temperature program began at 70 °C (2 min), raised to 150 °C at 25 °C/min, ramped to 200 °C for 5 min at 5 °C/min, was further increased to 270 °C (2 min) at 5 °C/min, and lastly 290 °C for 5 min at 5 °C/min"	[325]KKUU
Honey	14	0.01–0.04	0.04–0.12	81.4–111.4	2.0–11.2	GC-MS/MS-TQ: "Splitless, initial temperature 70 °C (held for 1 min), raised to 160 °C at 10 °C/min, then raised to 240 °C at 2 °C/min, and finally increased to 280 °C at 20 °C/min (held for 6 min)"	[326]
Honey bees, bee bread, honey (HBH)	18	IDL: 0.015–0.33; MDL: (HBH): 0.015–0.165;		>90		GC-ECD: "The oven temperature program was: 100 °C held for 1 min, followed by an	[327]

Table 4 (*continued*)

MATRIX	No. OF COMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/ INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
		soil&flower: 0.006–0.066				increase of 5 °C/min to 150 °C, held for 1 min, increase 1.5 °C/ min to 240 °C, and then 10 °C/min to 300 °C, held for 10 min"	
Low density poly ethylene sheet	22	0.00–0.31		57%-104		GS Micromass MS-MS: "Samples were held at 140 °C for 2 min, ramped up to 180 °C at 10 °C/min, ramped up to 220 °C at 3 °C/ min, and ramped up to 315 °C at 10 °C/min and held for 10 min"	[328]

[a] = values not converted.

frequently been discrepancies in the Analytical Chemistry community. Likewise, there are numerous ways to estimate it as shown (Table 3); consequently, the figures obtained from numerous studies vary. Three alternative techniques, including S/N, calibration curve slope (CCS), and laboratory fortified blank (LFB), were used to estimate the LOD and LOQ and consequently LFB showed a lower value [18]. The suggested values for LOD and LOQ are 3σ and 10σ, respectively [17], however S/N greater than 3 and 10 for LOD and LOQ respectively, are rampantly used [19,52,257]. Instrument detection limits (IDLs) identified as 3(S/N) were substituted for substances that were not detected in the blank samples. If a certain compound was detected in the samples but its concentration was less than its MDL/IDL, it was deemed to be zero [119]. Furthermore, concentration less than LOD was assumed LOD/2 [105,257,260–262]. Likewise LOD/$\sqrt{2}$ [263], (LOD/2)$^{1/2}$ [264], LOD/2 or LOQ/2 were used for values < LOD and LOQ respectively, only when frequency of occurrence was 60 % or more [265], and if no value was detected, the value is deemed zero [266]. Also, congeners with the highest LOD was used as the LOD [267]. In cases where more than 50 % of the data concentrations are < MDL, I/2 MDL was used; although 70 % data courage is preferred [104]. IDLs were taken to be MDL*$\sqrt{2}/2$ for values whose MDL were not detected [232], and detection limit (DL) assumed as 4 blanks + 3*SD of the blanks in cases where a blank signal was observed [73]. A low LOD is preferred since it indicates that the analytical method is sensitive enough to detect small quantities or amounts of the analyte being tested [268]. This may reduce the applicability of analytical method in some situations. From Table 4, very high LOD and LOQ (60–720 ng/g and 30–2400 ng/g) were observed from tea [269], (10–100 ng/mL and 100–10000 ng/mL) [69], (12–987 ng/g and 40–3290 ng/g) from fruits and vegetables [270], and (3700–4800 and 12–16000 ng/mL) [271], (180–780 ng/g and 62–2380 ng/g) [272] from human milk (Table 5), respectively. Similarly, Serum of a leopard also showed high LOD (800–34000 ng/mL) [25], (150–12730 ng/mL) [273], (Table 5). Furthermore, a very low detection limits (3×10^{-9} – 7.6×10^{-5} ng/mL) in aqueous sample [243], (2.8×10^{-9} - 5.64×10^{-8} ng/mL) seawater [239], and (1×10^{-7} - 1.4×10^{-6} ng/m^3) air [104] were observed (Table 2). From both theoretical and experimental perspectives, there is a performance parameter that is highly debatable due to a lack of general knowledge and significant discrepancies in nomenclature and calculation methods [79]. Therefore, the values will vary as a result of the various ways that LOD and LOQ are determined.

3.6. Instrument method and optimization/method validation

Instrument factors such as sensitivity, selectivity, linearity, precision, accuracy, and calibration can affect the accuracy and precision of analyte concentration. Optimization of these parameters is necessary for obtaining the most precise results. The choice of the best instrument-optimized technique for determining analyte concentration depends on several factors such as the nature of the analyte, sample concentration, the matrix of the sample, purity of the solvent, the required sensitivity, and the desired level of accuracy and precision [18,416]. Frequently used instruments and techniques have been elaborated in this section. In order to isolate and identify specific chemicals based on their vaporization characteristics, gas chromatography (GC) is frequently employed to evaluate volatile OCPs [67]. GC-ECD is frequently used due to its excellent resolution and affordability [189]. However, a high-temperature GC analysis may not be suitable for OCPs due to the potential for thermal degradation or insufficient separation, and thus temperature optimization is critical [417]. It is important to start with a low or potentially cooling initial GC column temperature to concentrate analytes at the top of the column [418]. Raising the temperature can generally improve the ability of GC to separate substances since it increases analyte volatility [418]. However, there is a maximum ECD temperature (300–350 ^{O}C) that can be increased without being relatively affected by column bleed, neither compromising the stability of the analytes, nor resulting in thermal decomposition [58]. GC-MS is a very sensitive and selective method for analyzing volatile and semi-volatile chemical molecules [419], offering extremely low detection limits [54,420]. However, it is not efficient for measuring thermally unstable compounds that cannot be processed at

Table 5
Quality control/assurance in human and animal tissues.

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Fish	11	1.0–3.0	3.3–10.0	82.11–115.7	Single fibre 4.1–7.6 Fibre to fibre:8.0–12.0	GC-ECD: "Split mode at a split ratio of 10, The oven temperature program started at 60 °C for 1 min, followed by being increased at 40 °C min− 1 to 172 °C, held at 172 °C for 2 min, increased at a rate of 1 °C min− 1 to 195 °C, raised at 30 °C min− 1 to 280 °C and then kept at 280 °C for 2 min"	[13]
Fish	10	0.1–0.6		Spiked sample: 88.4 to 98.5		GC-MS: "The oven temperature was programmed as follows: 100 °C for 4 min, 7 °C/min to 310 °C, and 6 min hold at 310 °C. A 2 Ml sample was injected in splitless mode with the split outlet opened after 1.0 min"	[274]
Fish	16		0.02–0.05	75–105		GC-ECD: "the initial oven temperature of 180 °C was increased by 7.5 °C min− 1 to 220 °C and held for 5 min, then heated to 250 °C at 20 °C min− 1, and held for 10 min"	[329]
Fish	9	0.3		64–100		GC-MS: "The column temperature was initially set at 70 °C. This temperature was maintained for 3 min, and increased to 150 °C at a rate of 25 °C/min, then increased to 200 °C at a rate of 3 °C/min and then to 280 °C at a rate of 20 °C/min and maintained for 6 min, then increased to 325 °C at a rate of 50 °C/min and held for 1 min"	[330]
fish	9	3×10^{-5}-5X10^{-4}	0.09–1.8	64–110		GC-MS: "Splitless. the oven temperature program was as follows: from 110 to 170 °C at 1.5 °C/min (held for 5 min), from 170 to 226 °C at 2 °C/min (held for 5 min), from 226 to 280 °C at 40 °C/min, and finally kept at 280 °C for 10 min"	[331]
Fish	15		0.1–0.7	75–92		GC-MS	[332]
Fish	20	0–0.008		0.024–0.833 range[a]		GC-ECD: "oven heating ramp to 120 °C (for 1 min) until 240 °C increasing at a rate of 4 °C min−1; EDC at 300 °C; injector at 260 °C"	[333]
Fish(krill)	20	0.11–1.4		Surrogate: 80.3–137.1		GC-QqQ-MS: "initial temperature at 70 °C (held for 1 min), raised to 160 °C at 10 °C/min, raised to 280 °C at 5 °C/min and held for 5 min. Then it was raised to 300 °C at 20 °C/min and held for 5 min"	[282]
Fish	8	0.01–0.43	0.02–1.3	72–115	<25	GC-MS: "temperature program 80 °C for 2.5 min; then 20 °C/min ramp to 180 °C followed by 5 °C/min ramp to 230 °C and 35 °C ramp to 300 °C (held for 7 min). Total run time was 26.5 min"	[52]
Fish		MDL: 0.001				GC-ECD: "oven temperature: 260 °C starting from 0 to 180 °C for 0.3 min and continued at 5 °C/min to 220 °C, held for 12 min, and continued at 5 °C/min to 260 °C"	[334]

Table 5 (*continued*)

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Fish	10	0.12	0.40	internal standards:78-95		GC-ECD: "The furnace temperature was programmed as follows: 70 °C held for 2 min, ramp at 25 °C/min to 180 °C, held for 1 min, and finally, ramp at 5 °C/min to 300 °C. RT 31.4"	[277]
Fish	9			Ave: 124		GC-MS: "the oven temperature program started at 40 °C which lasted for 2 min, then was increased in turn at 10, 5, and 10 °C/min to 100, 260 and 300 °C respectively, and kept for 10 min"	[335]
Fish	18	0.06–0.45	0.25–1.45	88.7–99.3	3.9–9.4	GC-MS RT: "10.145–20.941 min"	[336]
Fish	8	0.042–0.179	0,14–0.60	75.6–124	0.61–3.96	GC-MS: "the oven program was set at 110 °C for 1 min, increased to 220 °C at 10 °C/min, to 300 °C at 5 °C/min where it was held for 10 min"	[337]
fish	14	2–6		75–144	<20	GC-MS/MS: "Splitless, the oven temperature program was as follows: 70 °C for 3 min, up to 150 °C at a rate of 50 °C/min, up to 200 °C at a rate of 3 °C/min maintained for 1 min, up to 280 °C at a rate of 20 °C/min maintained for 5 min and finally, up to 310 °C at a rate of 40 °C/min maintained for 4 min"	[287]
Lipids for Fish muscle	7	0.01		59–88	4–15	GC-FID	[338]
Fish muscle	7	0.01		79–89		GC-ECD	[339]
Fish muscle	9	0.1–0.7		92.6–101.4		GC-MS: "The oven temperature was programmed as follows: 100 °C for 4 min, 7 °C/min to 310 °C, and 6 min hold at 310 °C"	[276]
Fish muscle	9			56–78	7–13	GC-MS, GC-ECD	[285]
Shellfish	14		5	95.11–102.17	3.57–10.6	GC-MS/MS: "Splitless, it was initially isothermal for 1 min, raised to 150 °C at 40 °C/min, and finally maintained at 300 °C for 8 min. RT 10.31–13.45"	[340]
shellfish	5	0.003–2.705	0.01–9.02	70–120	<10	GC-MS/MS: "The oven temperature was programmed at 80 °C for 1 min, increased to 150 °C at a rate of 20 °C/min, followed by 5 °C/min ramp to 300 °C (held for 5 min) for the total run time of 39.5 min"	[341]
Shellfish, cephalopods	10	Shellfish: 0.10–0.80; cephalopods: 0.21–0.77	Shellfish: 0.31–2.41; cephalopods: 0.63–2.33	Shellfish: 83.5–117.4; cephalopod: 79.8–118.4	Shellfish: 0.3–27.5; cephalopod: 1.2–27.9	GC-MS/MS: "The oven temperature program was held at 60 °C for 2 min, initially. Then, the temperature was elevated to 165 °C at a rate of 30 °C/min, then increased to 195 °C at a rate of 15 °C/min (held for 1 min), increased from 195 to 210 °C at a rate of 2 °C/min, then elevated to 220 °C at a rate of 5 °C/min and then programmed to 300 °C at 10 °C/min, held for 1.5 min"	[342]
Swordfish	7	Muscle: 4×10^{-5}-0.02; liver: 2×10^{-5}-0.014; gonad: 1×10^{-5}- 0.016		49–8	6–28	GC-HRMS, UPLC-MS/MS	[343]

Table 5 (*continued*)

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Lipids of fish	21		0.1–0.5	65–114	3–19	GC-MS/MS: "50 °C (2.9 min); 30 °C.min−1 to 240 °C; 2 °C.min−1 to 270 °C; and 40 °C. min−1 to 340 °C (12 min)"	[74]
Lipids from fish	9	Ave: 0.01	0.01–0.06	Spiked standards:69.8–98.9; Internal standard: 84.1–98.3.		GC-MS: "oven temperature: start from 90 °C (0.5 min), increase 7 °C/min; 220 °C (12 min), increase 6 °C/min; 285 °C (7 min), increase 5 °C/min; and 295 °C (6 min) (post run)"	[344]
fish, crab, shrimp, shellfish, and turtle, sediment	5	0.3–1.5		75–11	6.6–17	GC-MS: "Column initial temperature 50 °C, hold for 3 min; 35 °C·min⁻¹ rise to 220 °C, hold for 1 min; 15 °C· min⁻¹ rise to 300 °C, maintain for 2 min"	[345]
Fish and crustacean	17	0.9–166		47–114		GC-HRMS	[26]
Water, sediment, Muscle of fish, crustacean, oft tissue of shellfish	8	Water: 0.250–2.240; 0.003–0.018; organism: 0.003–0.040		69–103	<12	GC-ECD: "Splitless, the temperature program initially commenced at 80 °C and increased by 20 °C/min up to 200 °C. It then increased to 250 °C at 4 °C/min and was maintained for 2 min; then, it was maintained at 280 °C for 5 min after increasing by 30 °C/min"	[346]
Tilapia muscle	24	0.006–0.087	0.020–0.289	Standard: 68.6–96.2; surrogate: matrix: 73.8–105.6	<17	GC-MS/MS: "The GC oven temperature was programmed as follows: 80 °C (5 min)→20 °C/ min→160 °C (0 min)→4 °C/min→240 °C (0 min)→10 °C/min→295 °C (2 min)"	[347]
Lipids of pacific salmon fish(muscles, egg, liver, male gonad)	10	0.1–0.6		Spiked samples: 92.6–101.4		GC-MS: "Splitless, the oven temperature was programmed as follows: 100 °C for 4 min, 7 °C/ min to 310 °C, and 6 min hold at 310 °C"	[98]
Muscles, gonad and liver of cockfish	12	0.005–0.547	0.017–1.825	Matrix: 78&85; surrogate: 90&110		GC-ECD: "Splitless, the oven temperature program was set with a 100 °C start, held for 1 min, followed by an increase of 5 °C/min up to 150 °C, held for 1 min, then 1.5 °C/min up to 240 °C, and then 10 °C/min up to 300 °C for 10 min"	[348]
Fish gonad	16			89–124	2–7	GC-MS: "The oven temperature was programmed to increase from 90 °C with 2 min hold to 180 °C with 2 min hold (25 °C/min), 220 °C with 2 min hold (1.5 °C/min), 275 °C with 2 min hold (3 °C/min), and final 300 °C with 4 min hold (25 °C/min)"	[349]
Muscle, liver, egg, male gonad of salmon	12	0.1–0.5		94.6–103.7		GC-MS	[350]
muscle, lung, liver, kidney, and blubber samples of dolphins	19	0.3–0.7		Internal standard: 107.3	<12	GC-MS: "The column oven temperature was programmed as follows: 70 °C held for 2 min, increased at 3 °C per minute to 270 °C and held for 5 min, and increased at 5 °C per minute to the final temperature of 300 °C and held for 10 min"	[351]
Brain &testes of dolphin	1	0.3–0.7		Spiked: 86.6–123.1; Surrogate: 71.63–128.9	<12	GC-MS: "The column oven temperature: 70 °C held for 2 min, increased at 3 °C per min to 270 °C and held for 5 min, and increased at 5 °C	[352]

Table 5 (*continued*)

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						per min to a final temperature of 300 °C and held for 10 min"	
Dolphin	19	Reporting limit: 0.11–25		50–150		:GC-MSD "the oven temperature was programmed from 90 °C (1 min hold) to 150 °C at 5 °C/min, to 260 °C at 3 °C/min, and to 320 °C at 20 °C/min (5 min hold)"	[284]
Liver and muscles of fish	29	Liver: 0.23–8.62; muscle: 0.09–3.45		Liver: 63& 86; muscle: 59&93		GC-MS: "The oven was programmed as follows: 60 °C for 1 min, then 40 °C/min until 120 °C, and finally 5 °C/min until 285 °C for 0 min (Total time: 35.5 min)"	[255]
Muscle and liver of fish	10	0.1–0.7	0.3–2.1	73–98.05	0.35–5.66	GC-MS: "The oven temperature was optimized and set as follows: 100 °C hold for 2 min, raised to 280 °C at 10 °C/min, and then hold for 45 min"	[353]
Liver, muscle of fish	8		Liver: 0.01; 0.03	90–99		GC-ECD, GC-MS/MS: "The temperature program of the column oven was initially 90 °C (3 min) to 215 °C (40 min) at 30 °C/min^{-1} and 275 °C (30 min) at 5 °C/min^{-1}; the injector temperature was 270 °C and the carrier gas was helium (1 mL/min)"	[354]
liver, gills, and muscles of fish	18	0.01–0.02	0.04–0.08	90.2–103	1.2–6.1	GC-ECD; GS-MS: ECD:"initial column temperature was 80 °C for 6 min, increased to 215 °C at a rate of 15 °C/min (hold for 1 min), then to 230 °C at 5 °C/min, and finally to 290 °C at 5 °C/min (hold for 2 min); MS: The separation temperature program was initially set at 85 °C for 0.3 min, increased to 150 °C (hold for 4 min) at a rate of 30 °C/min, then to 185 °C at a rate of 2 °C/min, and finally to 290 °C (hold for 5 min) at a rate of 4 °C/min"	[355]
Muscles of fish and prawns	16	0.001–0.017		86–135		GC-MS	[356]
"ater, sediment, lipids from fish	18	Water: 4×10^{-5}-4.56 × 10^{-3}; sediment: 0.001–0.124; fish: 0.005–0.547	1.4×10^{-4}-0.01522, sediment: 0.004–415	Matrix 78 and 85; spiked surrogate: 90 and 110; fish: 0.017–1.825		GC-ECD: "The oven temperature program started at 100 °C, held for 1 min, followed by an increase of 5 °C/min up to 150 °C, held for 1 min, followed by an increase of 1.5 °C/min up to 240 °C, and then by an increase of 10 °C/min up to 300 °C, and held for 10 min"	[283]
Lipid of fish and sediment	21	Lipids: 0.7–1.7; sediment: 0.42–3.2		Sediment: 68–115; fish: 74–109		GC X GC-TOFMS	[357]
shrimp	17			79.7–94.1	7.65–16.58	GC-MS: "The oven temperature was kept at 90 °C for 30s, then increased to 280 °C @ 8 °C min− 1 and then to 300 °C @ 15 °C min− 1 for 2.5 min. The split and carrier gas flow were 50 and 1 mL/min, respectively"	[358]
Zooplanktons, fishes, shrimps, water	16	0.1 and 0.5		72–108	7–15	GC-MS: "The GC oven temperature was programmed to hold at 80 °C for 1 min and raise the temperature to 180 °C at a rate of 15 °C/min. Once the GC oven attained 180 °C,	[359]

Table 5 (*continued*)

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						the temperature was increased to 205 °C at a rate of 3 °C/min and held for 5 min. At a rate of 40 °C increase min^{-1}, the oven temperature finally increased to 265 °C and was held for 20 min"	
Water, shrimps	14	MDL: Water: 0.006–0.013; shrimps: 0.95–2.89	MQL: Water: 0.020–0.044; shrimps: 3.16–9.63	Water: 60–88; shrimps: 56-96	2.9–13.9	GC-ECD/GC-MS/MS: RT: 16.41–25.19 min	[360]
Blubber (Seal)		0–0.04		85.5–99		GC-ECD	[361]
lichen		4.36		56–91		GC-ECNIMS	[362]
Marine mammals	19	0.3–1.2		Surrogate: 71–96; matrix: 93-103	<20	GC-MS/MS: "Splitless, initial oven temperature of 100 °C (1 min), ramping at a rate of 11.0 °C/min to 180 °C, then to 260 °C (0.00 min) at 3.0 °C/min, and finally at 20.0 °C/min to a final temperature of 300 °C with a final holding time of 6.00 min"	[363]
Plasma of blue-footed booby	19	7.5&7.8	22.5&23.3			GC-ECD: "The initial oven temperature was 150 °C. Two temperature gradients were programmed: the first from 5 to 245 °C/min and the second from 10 to 310 °C/min for 5 min"	[364]
	22			Surrogate: 66&72		GC-MS/MS: "The GC oven temperature was set at 100 °C for 0.5 min, then 20 °C/min to 160 °C, 4 °C/min to 290 °C, and finally 10 °C/min to 300 °C, and hold for 10 min"	[365]
shark	10		0.0001–0.0274	42–115		GC-HRMS	[257]
Plasma of sea turtles	15		0.0050	Spiked blank: 65.6–117; matrice: 62.5–120; internal standard: 53.6–90.8		GC-MS/MS: "The oven temperature program was 50 °C for 1 min, followed by an increase at 20 °C/min to 200 °C and an increase at a rate of 10 °C/min until it reached 300 °C, remaining constant for 5 min"	[366]
Green sea turtle	20			Liver: 81–108; muscle: 56-82		GC-MS: "Splitless, GC column oven temperature was initially at 60 °C held for 1 min, then increased at a rate of 25 °C/min to 160.0 °C. The temperature was then increased to 240.0 °C at a rate of 4.0 °C/min and then finally to 290.0 °C at a rate of 10.0 °C/min where the temperature was held for 11 min"	[367]
Blood plasma of green sea turtle	11	0.18		100.86–119.14		GC-MS: "The oven temperature profile was programmed from 45 °C to 150 °C at 20 °C/min, and then to 300 °C at 2.5 °C/min"	[267]
Feces, commercial feed, soil of primates	OCPS(21), CUP (29)			CUP: 63-130	OCP: 28–54; OCP: 10-22	OCP: GC-EC:D "The GC oven temperature program was as follows: initial 100 32 °C for 1 min, 1 °C/min to 240 °C, 10 °C/min to 280 °C, and held for 20 min, 80 °C for 0.1 min, 500 °C/min to 280 °C, and held for a final 20 min". CUP: GC-MS "The GC oven temperature program was as follows: initial 80 °C for 2 min, 30 °C/min to 150 °C, 2 °C/min to 240 °C,	[368]

Table 5 (*continued*)

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						20 °C/min to 300 °C, and held for 5 min. The MS transfer line was held at 280 °C, and the ion source and quadrupole temperatures were both held at 150 °C″	
Crab	10	0.03–1.08	0.1–3.6	80.2–119.8	1.4–12.9	GC-MS: "The oven conditions were set as follows: 80 °C hold for 1 min, 80–170 °C at 30 °C/min, then 170–240 °C at 5 °C/min, hold for 1 min, finally 240–300 °C at 20 °C/min"	[369]
Mussels	4	0.01		60115	3–20	GC-ECD; GC-MS: "the column temperature programme was 90 °C (3 min) to 215 °C (40 min) at 30 °C/min and to 275 °C (30 min) at 5 °C/min"	[370]
Mussels	14	0.02–0.11	0.16–0.8	61–120	0.7–7.0	GC-MS: "Splitless, the temperature program was as follows: 1 min isothermal program at 90 °C, increased to 180 °C at 30 °C/min, to 300 °C at 5 °C/min and held for 1 min"	[371]
Mussels		0.5	<500	80–120		GC-MS/MS: "GC oven was temperature programmed from 80 °C (held for 2 min) to 300 °C at 12 °C.min^{-1} and then held for 5 min"	[372]
mussel	23	0.01–0.058	0.028–0.93	99.6–106	1.14–6.60	GC-MS/MS: "The oven temperature programme began at 70 °C, increased to 240 °C at 20 °C/min, and subsequently increased to the final temperature of 310 °C at 8 °C/min (held for 11 min)"	[373]
Mussel		0.05–0.09		Surrogte: 83.3, 80.5		GC-ECD: "70 °C/2 min to 260 °C at 3 °C/min, and then held for 25 min"	[374]
Frog	22		0.13–4	SRM: 75–110:	<12	GC-ECD: "Splitless, the oven program was initialised at 100 °C held for 1 min, ramped at 12 °C/min to 180 °C, ramped at 4 °C/min to 240 °C, ramped 10 °C/min to 270 °C and held for 5 min"	[24]
Bivalves	10	0.1–0.6		92.6 to 101.4		GC-MS/GC-ECD: "The oven temperature was programmed as follows: 100 °C for 4 min, 7 °C/min to 310 °C, and 6 min hold at 310 °C"	[31]
Bird tissue	9	0.3	0.9	74–103	<14	GC-MS: "The column temperature was initially set at 70 °C. This temperature was maintained for 3 min, and increased to 150 °C at a rate of 25 °C/min, then increased to 200 °C at a rate of 3 °C/min and then to 280 °C at a rate of 20 °C/min and maintained for 6 min, then increased to 325 °C at a rate of 50 °C/min and held for 1 min"	[330]
Muscle, whole of seabirds	20	IDL: 0.055–0.903	0.0–0.772	50–120	<15	GC-HRMS	[254]
Tissue, liver, brain of bird	13	1		91–102		GC-ECD: "oven temperature was programmed as 180 °C 3 min; 4 °C/min 260 °C 15 min"	[375]
Feathers of bird	8		0.02	90–100		GC-ECD: "2 min at 60 °C, gradual heating from 60 to 160 °C at the rate of 20 °C/min; 3 min at 160 °C, gradual heating from 160 to 280 °C at	[256]

(*continued on next page*)

Table 5 (*continued*)

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						the rate of 2.5 °C/min; 10 min at 280 °C, gradual heating from 280 to 300 °C at the rate of 20 °C/min; and 10 min at 300 °C"	
Feathers of bird	23	0.027	0.05	97.5	4.27	GC-MS	[376]
Feathers of pigeon	25	0.006–1.3	0.3–3	61–120	1–18	GC-QqQ-MS	[377]
Feathers of penguins	6	0.011–2.67		Internal std: 69		GC-MS/MS: "Splitless, the column temperature was initially held at 70C for 2 min, raised to 150C at the rate of 25C/min, then to 200C at the rate of 3C/min, and to 280 at a rate of 8C, held for 10 min, finally to 300C at a rate of 100C/min, and held at the final temperature for 5 min. Total analysis time was 47.067 min"	[288]
Penguin tissues	31		2.2–4.6	Sur: 53–136; spike: 113; SRM: 130		GC-ECD: "The column oven was programmed for an initial temperature of 100 °C for 1 min and a rate of 5 °C/min to 140 °C, then held for 1 min at 140 °C, ramped to 250 °C at a rate of 1.5 °C/min, held for 1 min and finally increased at a rate of 10 °C/min to 300 °C and held for 5 min"	[378]
Blood of penguins, short-tailedshearwaters	14	MDL: 0.04–0.95	MQL: 0.12–3.2	82–115		GC-MS/MS: "The initial inlet temperature was 90 °C for 0.1 min before ramping to 325 °C at 900 °C/min. The oven temperature was 50 °C for 1 min and then ramped to 320 °C at 25 °C/min with a hold time of 4 min"	[22]
Eagle-owl feathers	12		0.05–0.10	Internal: 71-92	<30	GC-MS: "Splitless, the temperature of the DB-5 column was programmed from 90 °C, kept for 1.5 min, then increased with 15 °C/min to 310 °C, kept for 15 min"	[73]
Feathers, liver of owl	16	0.03–0.54		46-146 (feathers) 86 146 (liver)		GC-MS	[379]
Blood cell and feather of Antarctic petrels	5	0.00172–0.305;	0.00517–1.016	45–104		GC-HRMS: "Splitless, the PTV injector was held at 90 °C for 0.1 min, ramped to 320 °C at 5 °C/min with a hold time of 5 min"	[380]
Pellets of black vulture		4–310		>89		GC-MS/MS: "The GC oven temperature was programmed as follows: initial temperature of 70 °C, hold at 70 °C for 2 min, increase the temperature to 300 °C at a rate of 20 °C/min, and hold at 300 °C for 8 min"	[381]
Hamster head	9	0.084–0.725		87.2–96.1; surrogate: 76.5&121.3		GC-MS: "started with the temperature of 100 °C, held it for 1 min, then increased 43 at 10 °C/min to 220 °C and 20 °C/min to 280 °C, held it for 10 min"	[382]
Liver of wild boar	4	0.008–0.05	0.02–1.1	86–120	3–18	GC-QqQ-MS/MS	[383]
Water, sediment and waterbird lung tissue	18	0.00001–0.0001 (water), 0.01–0.05 (sediment) (plant) 0.05–0.64 bird.		Spiked: 74–102; surrogate: 70&82	3.6–11.4	GC-ECD: "The oven was programmed for an initial temperature of 100 °C, increased to 200 °C at 4 °C/min, then increased to 230 °C at 2 °C/min, and raised to 280 °C at 8 °C/min"	[77]
Bird's liver and lung	10	Lung:2.59, Liver: 0.689; fat tissue: 0.309		50-150; (Matrix spike): 78.1–100; 59.0 (surrogate_	<20	GC-MS: "Splitless, oven temperature was set at 80 °C for 1 min, ramped to 240 °C at 20 °C/	[92]

Table 5 (*continued*)

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						min, kept at 240 °C for 6 min, then heated to 280 °C 10 °C/min, and held at this temperature for 10 min.RT 11.7O-13.55"	
Bird's egg	10	0.5		94–105		GC-ECD: "oven temperature was programmed as 180 °C-3 min; 4 °C/min-260 °C-15 min"	[384]
Chicken Brain	9	0,01–0.40		96–125		GC-ECD and LRMS	[385]
Lipid material, homogenates of muscle, liver and gonad	19	0.005–0.547	0.017–1.825	>90		GC-ECD: "The oven temperature program started at 100 °C, held for 1 min, followed by an increase of 5 °C/min up to 150 °C, held for 1 min, followed by an increase of 1.5 °C/min up to 240 °C, and then by an increase of 10 °C/min up to 300 °C, and held for 10 min"	[386]
Muscle of crocodile	6	0.04–0.16		75–110		GC-ECD: "The GC oven temperatures were programmed as follows: 100 °C for a 1 min hold; then temperature was ramped at 12 °C.min^{-1} to 180 °C; followed by an increase of 4 °C.min^{-1} to 240 °C and then by 10 °C.min^{-1} to 270 °C"	[387]
Serum of leopard	22	800–3.4 × 10^4		78.8	<11	GC-ECD: "Splitless, the oven program initiated at 100 °C held for 1 min, followed by a ramp of 20 °C/min to 200 °C, then changing to a ramp of 6 °C/min until 260 °C held for 4 min"	[25]
Cow and human milk	50	0.15–0.9	0.15–3	74–121	1–18	UHPLC-MS, GC-ECD: primary temperature, 100 °C held for 1min, then increased with the rate of 3 °C/min to 230 °C. Total run time: 44.33 min.	[388]
Buffaloes and cow's milk	18	0.12–1.54	0.42–5.14	Surrogate: 75&84; spiked: 83–130	<20	GC-MS: "for the first 3 min the temperature was 150 °C, 4 °C/min to 290 °C, and the isothermal process was kept for 10min"	[389]
Donkey milk	7	0.01–0.06	0.05–0.3	81–105 %	<15 %	"The oven temperature was programmed as follows: 110 °C for 1 min, 8 °C/min to 230 °C, hold for 8 min at 230 °C, then 3 °C/min to 300 °C and hold for 6 min at 300 °C"	[390]
Breastmilk	8	180–780	62–2380	52.5–117.5	1.61–12.81	GC-ECD: "The initial temperature was 80 °C, ramp at 30 °C min-1 to 180 °C, ramp at 3 °C min-1 to 205 °C, held for 4 min, ramp at 20 °C min-1 to 290 °C, held for 8 min, ramp at 50 °C min-1 to 325 °C. Deltamethrin: the oven temperature was maintained initially at 130°c, held for 1 min, ramp at 30 °C min-1 to 280 °C, held for 16 min and ramp at 50 °C min-1 to 325 °C, held for 3 min. Total time: 27.92 min"	[272]
Human milk	27	150–12730		76–116.2	1.09–15.4	GC-MS: "initial temperature was set at 70 °C and held for 2 min, increased at 25 °C/min to 150 °C, then immediately increased to 200 °C at a rate of 3 °C/min, finally increased at 8 °C/min to 290 °C, held for 6 min"	[273]
Human milk	18	1.7–4.3	5.8–43.1	64.7–128.2		GC-MS: "The oven heating program was started at 100.0C (4.00 min), increased from 15.0C	[391]

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Table 5 (*continued*)

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						min1to 200.0C (0.5 min), at 2.0C min1230.0C (3.0 min), and ending with 15.0C min1to 280.0C, with total time of 27.5 min"	
Milk	8	3700–4800	1.2×10^4-1.6×10^4	93–97	<10	GC-ECD, GC-MS: "The oven program was operated in 6 steps: (1) the starting temperature of the column oven was 40 °C, and this temperature was maintained for 1 min; (2) the temperature was increased to 120 °C at a rate of 20 °C/min, where this temperature was maintained again for 1 min; (3) subsequently, the temperature of the oven was increased to 150 °C at a rate of 10 °C/min, hold 1 min, (4) next at 10 °C/min to 180 °C, hold 1 min; (5) next at 20 °C/min to 200 °C, hold 1 min and (6) finally at 10 °C/min to 290 °C, where it was kept for 2 min (the total running time was 25 min)"	[271]
Breastmilk	8	<0.2		>90	<15	GC-ECD: "initial oven temperature of 150 °C held for 1 min, ramped at 3 °C/min to 200 °C and then ramped at 8 °C/min to 280 °C held for 10 min"	[392]
breastmilk	20	0.01	0.01–0.4	69.9–101.3		GC-MS: "oven temperature—start from 90 °C (0.5 min), increase 7 °C/min, 220 °C (12 min), increase 6 °C/min, 285 °C (7 min), increase 5 °C/min, 295 °C (6 min)"	[393]
Water, milk, tea	8			91–108	2.4–9.9	GC-ECD, GC-MS: "The GC oven temperature program was started at 110 °C and held for 1 min, then increased up to 310 °C at 15 °C/min and held for 1 min. RT 7.30–10.7 min"	[394]
Tap and well water, tea, milk	5	0.002–0.08	0.005–0.26	Tap and well: 84.1–100.9; green tea and milk: 83.4–101.6	Tap& well: 8.2; green tea& milk: 1.4–8.6	GC-MS "MRM, the GC oven temperature was set as follows: 70 °C for 1 min, 10 °C min−1 to 180 °C (held for 5 min), 5 °C min−1 to 220 °C, and 30 °C min−1 to 280 °C (held for 8 min)"	[395]
Milk, soil, water	17	0.11 to 0.83	0.37–2.75	23.35–100.17		GC-ECD: "Splitless, the initial column temperature was 75 °C that was maintained for 1 min, increased at a rate of 25 °C/min to 150 °C, raised at a rate of 6 °C/min to 225 °C, and lastly increased at a rate of 15 °C/min to 290 °C where it was maintained for 10 min. The total analysis time was 31 min"	[396]
Human Serum	28	22.11–128.66		78–97	1.8–13.3	GC-HRMS: SIM	[40]
Lipid serum of human	17	0.84–25.7	3.20–89.9	86–120		GC-MS/MS	[41]
Blood	11	0.01–0.5 L		Spiked blanks: 93–106, matrix spikes: 94–112 procedural blanks, Surrogate: 63–91		GC-MS: "Splitless mode, column flow rate was set at 1 Ml min− 1 in multiple reaction monitoring mode, with the starting oven temperature at 100 °C (1 min), ramping at 11 °C min− 1 to 180 °C, then 3 ∘Cmin-1 to 260 °C and ultimately to 300 °C at rate of 20 °C min− 1 with final holding time of 6 min"	[397]

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Table 5 (*continued*)

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Blood	4	MDL:0.37–1.8	MQL: 0.11–0.53	82–116			[398]
Blood	9	2–5	5–40			GC-MS/MS	[399]
Serum of man	15	MDLs: 0.0113–0.0318			>20	GC-MS/MS: Splitless,	[400]
Blood of man; drinking water	8	0.05 pg[a]		95		GC-ECD	[401]
Blood(serum) of man	18	10–210		76.0–130.5		GC-MS	[43]
Semen, Blood(serum) of man	18	0.01–0.21		76.0–130.5		GC-MS	[42]
Blood serum of women	17	0.004–0.11	0.02–0.38	85–115	4–20	GC-MS/MS: "The oven temperature program was as follows: 70 °C for 3 min, up to 150 °C at a rate of 50 °C min−1, up to 200 °C at a rate of 3 °C min−1 maintained for 1 min, up to 280 °C at a rate of 20 °C min−1 maintained for 5 min and finally, up to 310 °C at a rate of 40 °C min−1 maintained for 4 min"	[402]
Serum of ovarian of human	9	0.002–0.015	0.005–0.04			GC-MS/MS	[403]
Serum from cord blood	4	0.012		88–94		GC-MS: "temperature was set on 110 °C for 2 min, then increased by 15 °C/min to 285 °C thus kept for 5 min, and finally increased by 5 °C/min to 300 °C and kept thus for 15 min"	[28]
Human Umbilical cord	20			3.35–90.07		GC-MS	[261]
Human umbilical cord blood serum	4	0.26–1.12	0.86–3.74	44.86–71.80	1.75–2.43	GC-MS: "The column temperature was programmed from 50 °C (3 min) to 150° at 30 °C/min, 150 °C to 300° at 10 °C/min (2 min). The carrier gas was helium (purity 99.99 %) with a flow rate of 1 Ml/min. RT: 15.27–22.29 min"	[404]
Plasma/serum	23	0.001–0.029	0.002–0.093	85.9–109	1.97–19.6	GC-MS/MS: "The oven temperature program was started at 70 °C, increased to 240 °C at 20 °C/min, and continuously increased to 310 °C at 8 °C/min and maintained at the final temperature for 11 min"	[405]
Plasma/serum	7)	0.02–0.07			4–40	GC-MS/MS: "The GC oven program was set to following values: 115 °C (2 min), 4 °C/min, 260 °C (1 min), 30 °C/min, 320 °C (2 min)"	[406]
serum	26	7×10^{-5}-0.01344		30–124		HRMS/HRMS	[407]
Blood serum	11			74–120	<20	GC-MS/MS, UHPLC–MS/MS	[101]
Human tissue (liver, kidney, heart, spleen, lung, brain and abdominal fat)	20	1.0–16		85–109	<12	GTC-MS: "Splitless, the GC oven program started with an isothermal stage at 100 °C for 0.5 min, increasing at 10 °C/min ~180 °C, and then at 2 °C/min ~225 °C (2 min), to finally reach 265 °C at a rate of 20 °C/min, which was maintained for 1 min"	[44]
Human blood	8	0.004; 5×10^{-5} ng[a] for detector		Ave: 95		GC-ECD	[408]
Human blood	4,4′DDE, HCB	0.03		Int. standards: 97.6, 75.62		GC-MS: "Splitless, 90 °C for 2 min followed by an increase of 20 °C/min to 160 °C, then 5 °C/min to 245 °C and held for 5 min, to finally be	[409]

Table 5 (*continued*)

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
						increased of 20 °C/min up to 300 °C, held 5 min"	
Human blood serum	19	0–0.022		60–130		GC-ECD: "The oven temperature was programmed from an initial temperature 80 °C for 1 min, and then increasing at 30 °C/min up to 160 (2 min hold) after that, increasing to 260 °C at a rate of 3 °C/min and was maintained at 260 °C for 12 min"	[410]
Serum and venous blood of human	19	0.01–0.21	0.03–0.08	76.0–130.5; intra/inter batch: 9.0–11.6; biomarkers: 87-102		GC-MS: "The oven temperature program was kept initial temperature at 40 °C for 1.5 min, followed by 25 °C/min to 90 °C for 1.5 min, 25 °C/min to 180 °C, 5 °C/min to 250 °C, and 50 °C/min to 300 °C for 6 min"	[411]
Human placenta	20	0.002–0.17	0.006–1.81	56–148		GC-MS: "Split, the column temperature raised from the initial temperature of 50 °C (hold for 2 min) to 150 °C (hold for 15 min) at 10 °C/min, then ramped to 240 °C (hold for 30 min) at 3 °C/min, and finally increased to 300 °C (hold for 6 min) at 10 °C/min"	[412]
Placenta sample	13	0.27–0.77	0.82–2.30	86.2–101.1	5.2–10.8	GC-MS: "Splitless, GC oven program was initiated at 70 °C (with a holding time of 2 min), following temperature increases of 25 °C/min to 150 °C, 5 °C/min to 200 °C (with a holding time of 5 min), 5 °C/min to 270 °C (with a holding time of 5 min), and 25 °C/min to 290 °C (with a holding time of 5 min. RT 44 min"	[280]
Hair	7	0.01–0.1	0.02–0.38	2.51–200.54		GC-MS: "The starting temperature was 60 °C, held for 1 min, ramped to190°Cat40 °C/min, ramped to 280°Cat5°C/min, held for2 minutes, ramped to 290°Cat10 °C/min and held for 5 min"	[72]
Hair	25	0.1–1.0	0.1–25.0	67–139	1–52	GC-MS/MS: "It was initially held at 60 °C for 1 min, then raised at a rate of 20 °C/min to 180 °C, held during 1 min. Afterwards, the temperature was increased by 4 °C/min to 240 °C and then by 60 °C/min to 300 °C"	[413]
Hunan Hair	14	0.011–1.38		Int std: 83-180		GC-MS: "The column temperature was initially held at 70 °C for 2 min, raised to 150 °C at the rate of 25 °C/min, then to 200 °C at the rate of 3 °C/min, and to 280 at a rate of 8C, held for 10 min, finally to 300 °C at the rate of 100 °C/min, held at final temperature for 5 min. Total analysis time was 47.067 min"	[289]
Hair, liver		Liver: 0.01–0.11; hair: 0.14–1.4	Liver:0.1–0.50; hair: 0.1–10	Liver: 74–119; hair: 61- 12	<20 %	GC-QqQ-MS/MS	[414]
Hair of wild cat	10		0.01–0.5	94–110	<10	GC-ECD: Splitless, "100 °C (1 min), 10 °C/min to 240 °C (1 min), 3 °C/min to 260 °C (1 min), 20 °C/min to 300 °C (10 min)"	[415]

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Table 5 (*continued*)

MATRIX	No. ofCOMPOUNDs	LOD	LOQ	RECOVERY	RSD	INSTRUMENT/INSTRUMENTAL CONDITIONS/TOTAL RUN TIME	REFERENCE
Hair	7	0.01–0.1	0.02–0.38	2.51–200.54		GC-MS: "The starting temperature was 60 °C, held for 1 min, ramped to190°Cat40 °C/min, ramped to 280°Cat5°C/min, held for2 minutes, ramped to 290°Cat10 °C/min and held for 5 min"	[72]

^a = values not converted.

high temperatures [421]. It is also challenging to analyze samples with relatively low OCPs concentrations by GC-MS due to higher method detection limit (MDLs) of OCPs by GC-MS compared to GC-ECD [152,171]. GC-ECD is highly selective for OCPs such as DDT, HCH, endosulfan, aldrin, and dieldrin [46]; because the electron capture detector only reacts to substances that contain electronegative functional groups like halogens [422], despite having low selectivity [423]. Albeit, MS/NCI gave better LODs and LOQs than ECD in a recent study [189]. Therefore, it is recommended to confirm GC-ECD analysis with GC-MS [131,152,238]. In food safety laboratories, gas chromatography coupled to tandem mass spectrometry (GC-MS/MS), a very sensitive method, is gradually replacing GC-MS since it offers a higher degree of separation on pollutants and overcome the complexity and low concentration being limited by the analysis [424]. For the purpose of capturing the presence of secondary and tertiary metabolites of OCPs in environmental and biota samples that are specifically toxic to human tissues and organs, more sophisticated measurement techniques such as gas chromatography combined with high-resolution mass spectrometry (GC-HRMS) and matrix-assisted laser desorption time of flight mass spectrometry (MALD-TOF MS), or GC-TOF are recommended [55]. Despite not being used as frequently for OCP analysis, liquid chromatography (LC) is an alternative, particularly for compounds with low volatility, strong polarity, and thermal instability [67]. High polarity and non-volatile and/or thermally labile pesticides, as well as those that are not GC-amenable, are only a few of the substances in plant-derived food that are quickly and effectively identified using the liquid chromatography-mass spectrometry (LC-MS) approach. However, because LC-MS often operated in selected reaction monitoring to detect target compounds, they are less desirable for tracing pesticides analysis [53]. Ultra-High-performance liquid chromatography coupled with quadrupole time of flight mass spectrometry (UHPLC-Q-TOF-MS) can be used as an alternative strategy to separate OCPs based on their polarity and chemical properties [57]. High-Performance Liquid Chromatography (HPLC) has also been used for OCPs analysis like imidacloprid [425], but there are fewer reports on its use. Tables 2, 4 and 5 show the different instrument conditions and run times used in OCPs analysis, with almost all the analysis being carried out with GC-MS and GC-ECD. The initial temperature ranges from 40 to 195 °C except for very extreme values 0 and 240 °C, to the maximum temperature ranging from 220 to 340 °C. Accurate analytical assessments of environmental samples are critical for wise decision-making in various areas of society [17].

3.7. Discrepancies in quality assurance

There is still a possibility of the discrepancies despite using the right instrumentation and extraction procedures. This is suggested by the multiple ways of carrying out the QC/QA of the analysis of not just the OCPs but other pollutants. Different methods used for the QC/QA have been shown previously (Tables 1 and 2), and these will give different results when applied with same methods extraction and instrumentation. Although, majority of the published research used procedural and spiked blank and 3 or 10 (S/N) for recoveries and detection limits respectively. Various extraction methods for OCPs from air such as soxhlet (Lee et al., 2022; Lohmann et al., 2021; Miglioranza et al., 2021), automatic extraction (Llanos et al., 2022), and ultrasound-assisted extraction micro scale (UAE-MSC) (Beristain-Montiel et al., 2020) were utilized. However, the method detection limit (MDL) varied. The lowest MDL values ($1 \times 10\text{-}7$ – $1.4 \times 10\text{-}6$ ng/m3) were observed with 3 times the standard deviation (3(SD)) (Lohmann et al., 2021), followed by $5 \times 10\text{-}5$-$1.03 \times 10\text{-}3$ using the average blank + 3(SD) (Miglioranza et al., 2021). Most studies did not provide information on the sample volumes used, making it challenging to determine the factors affecting the limit of detection (LOD). For water samples (Table 2), liquid-liquid extraction (LLE) yielded a relatively lower LOD of 0.000002–0.00003 ng/g (Necibi and Mzoughi, 2020) and 0.000006–0.00003 ng/mL (Sah et al., 2020) with a sample volume of 1 L, compared to solid-phase extraction (SPE) with LODs of 0.001–0.01 ng/mL (Yang et al., 2021) using a smaller sample volume of 200 mL, and 0.0006–0.003 ng/mL with a 1 L sample (Behrooz et al., 2020). Additionally, dispersive liquid-liquid microextraction (DLLME) achieved a very low LOD (0.00006–0.003 ng/mL) with a smaller volume of 10 mL (Carvalho et al., 2020). Internal ultrasonic technology which resulted in a relatively lower LOD ($3 \times 10\text{-}9$-$7.6 \times 10\text{-}5$ ng/mL) (Zhang et al., 2022), and ($2.8 \times 10\text{-}9$ – $5.64 \times 10\text{-}8$ ng/mL) (Kang et al., 2022) were also obtained. The choice of sample volume becomes a challenge when considering that low LODs were achieved with both smaller and larger volumes. It is generally recommended to use small sample quantities for OCP level measurements (Fernandes et al., 2012). Analytical procedures that require less initial sample volume and/or extraction solvent includes SPE procedures which enables significant volume reductions in both the extracting solvents and aqueous sample quantities (Farahani et al., 2008). For soil samples, a 10 g sample size was analyzed using the Soxhlet extraction method with chromatographic cleanup (Ding et al., 2022; Emoyan et al., 2022; Nyihirani et al., 2022), except for the use of florisil SPE cartridges (Khuman et al., 2022). However, a relatively lower MDL (0.00011–0.00549 ng/g) was obtained using the average blank + 3 (SD) (Ding et al., 2022). SPE was also employed for soil samples (1 g), resulting in LOD as low as 0.002–0.05 ng/g (Tadesse, 2021). The best approach, which has lower limiting values for the Soxhlet and solid phase extraction of OCPs from sediment and water, respectively, is the laboratory enriched blank [18]. Human tissue analysis (Table 5) (Belda et al., 2021; Pi et al., 2020; Yurdakok-Dikmen et al., 2022) revealed that the smallest sample size (0.2 g) extracted using salting-out liquid-liquid extraction (SALLE) combined with dispersive liquid-liquid extraction (DLLME), despite being environmental friendly, allows extraction, cleanup, and preconcentration, resulted in the relatively high LOD (1.0–16.0) and relatively lower recovery (85–109 %) (Belda et al., 2021). The centrifuge method was used for the extraction of 2 mL serum samples (Kaya et al., 2022; Varakina et al., 2021) and demonstrated a relatively lower LOD (0.004–0.11 ng/g) using 3 times the standard deviation (3(SD)) of the procedural blank and higher recovery (85–115 %) (Varakina et al., 2021). Ultrasonication showed relatively lower LOD (0.12–1.54 ng/mL and 0.01–0.06 ng/mL) (Monnolo et al., 2020; Sana et al., 2021) in the extraction of animal milk compared to the values (180–780 ng/mL and 150–12730 ng/mL) obtained from the QuEChERS method (Kuang et al., 2020; Mekonen et al., 2021) and GDME (3700–4800 ng/mL) (Lobato et al., 2021). For the extraction of fish, the use of a soxhlet extractor and SPE cleanup (Ma et al., 2020), chromatographic column (Jeong et al., 2020), and separating funnel (Donets et al., 2021) resulted in an LOD of 0.1–0.7 ng/g with a sample size of 0.4 g. Although the same calculation (signal-to-noise ratio multiplied by 3) was applied to the SPME and centrifuge method (Zang et al., 2023) and QuEChERS

(Habibullah-Al-Mamun et al., 2022), the latter yielded a lower LOD (0.01–0.43 ng/g) with a larger sample size of 10 g. Lipids are the primary interference factors in fish tissues and can be extracted concurrently with the target analytes [426], thus needs to be removed to reduce interferences [427,428]. Albeit, a study found that the detection limits attained using SPME-GC-MS are generally better than those attained following a Soxhlet extraction [429]. The ultrasonication method was also used to extract 1 g (Table 4) (Li et al., 2022) and 2 g (Zang et al., 2021) of fruits and vegetables using an LOD based on a 3:1 signal-to-noise ratio. Lower LODs (0.01–0.20 ng/g) were obtained with a sample size of 2 g. A sample size of 5 g yielded a low LOD (0.0001–0.0004 ng/g) with the QuEChERS method (Adeyi et al., 2021), whereas the 2 g sample (Yu et al., 2021) and 10 g sample gave higher LODs (12–987 ng/g) (Siraj and Ejeta, 2022) and (10–100 ng/mL) (Collimore and Bent, 2020), respectively.

3.8. Method validation and optimization

Method validation is a method to ensure that the analytical approach used for a particular test is appropriate for its intended use [430,431]. A procedure has not been shown to produce reliable data if the method validation has not been carried out or has been carried out insufficiently [432]. Design methodology integrate the knowledge of professionals in the processes and equipment that enable design and product development [433]. In design research, the validation of design methodologies continues to be a challenging and developing process [434]. However, Support for the evaluation of design technique applicability is currently lacking [433]. To ensure a high level of understanding and promoting simplicity of use, it is anticipated that the more intricate the technique, the more effort will be necessary for method introduction [433]. It is crucial to correctly identify the test method that was utilized, which may determine how parameters affect the effectiveness of a procedure before relying on the data gathered to characterize the product [435].

The link between known concentrations of the analyte in the sample and the instrument's response is represented by analytical calibration [79]. The ideal validated technique is one that has fully advanced through a joint study in compliance with internationally harmonised guidelines for the design, conduct, and interpretation of method performance studies [436]. The OCPs are typically present in low concentrations, hence high-quality sample preparation techniques are needed to help achieve the desired LOD and LOQ, get rid of any potential interferences from a complicated matrix, and accomplish the target analyte enrichment before instrumental analysis [437]. Matrix-matched calibration curves are used to avoid any potential matrix effects and to obtain accurate data from the samples that have been tested [438]. When a calibration curve is designed with the wrong concentration range, it could result in a false negative result. Although they can be analyzed, low-concentration values are not included. Near background levels, there is an increased likelihood of concentration overestimation [439]. Generally, matrix effects as documented by SANTE/11813/2017 are offset using matrix-matched calibration. Use of standard addition or internally labelled isotope standards is the most efficient method of mitigating matrix effects. Moreover, the reporting limit's (RL) lowest calibration level (LCL) must be equivalent to or lower than that level. No lower limit than the LOQ may exist for the RL [440].

Validation experiments, as stated in document SANTE/11813/2017, showed that the acceptance criteria for method validation of OCP analytes include ≤20 % for high precision, r > 0.995 for linearity and 70–120 % for recovery [177,189,440]. However, recovery rate outside this range can be accepted if they are consistent (≤20 %) [440].

One-factor-at-a-time (OFAT) and the Response Surface Methodology (RSM) are two methods for optimizing process variables. Compared to the time-consuming traditional OFAT approach, the RSM method is one of the most effective methods for optimizing process variables since it assures that the entire design of experiments is accomplished in a more effective manner [441,442]. RSM is a set of statistical and mathematical methods for optimizing the influence of process factors in experiments and is based on the design of experiments (DOE) [443].

Factorial design is another experimental approach which is the difference between the mean value of all the measurements in the factor's maximum and minimum [371]. The use of a factorial design also enables the examination of the effects of several variables that may affect a response (such as the proportion of pesticides recovered) [371].

High sensitivity, high resolution, and symmetrical peak morphologies (no tailing) can be achieved by optimizing the chromatographic settings across a number of trials for the separation and detection of OCPs [355]. The method should be kept under regular evaluation and updated as necessary to reflect advancements in technology, legislation, or lab specifications. Instrument performance, the reagent quality, and the consumables should be monitored to ensure repeatable and trustworthy results.

4. Conclusion, limitation, and future perspectives

The significance of optimizing analytical techniques to increase sensitivity, selectivity, and effectiveness was discussed in this review. Using cutting-edge equipment and methods, such as GC-MS and GC-ECD, this can be accomplished. To ensure that the analytical techniques utilized for OCP analysis are reliable, accurate, and repeatable, standardized validation methodologies are required. This is especially crucial considering the potential health concerns associated with exposure to OCPs and the requirement for precise risk evaluations to guide policy decisions. Knowledge gap and future perspective that should be considered for additional research should include greater emphasis on environmental and clinical applications. With growing knowledge of how environmental pollutants affect human health, there will probably be more emphasis on the analysis of OCPs in environmental and clinical samples. As a result, it is likely that research in this area will continue to grow and that new techniques will be created and improved to suit the requirements of this field.

Data availability statement

Data included in article/supp. material/referenced in article.

CRediT authorship contribution statement

Chinemerem Ruth Ohoro: Conceptualization, Data curation, Investigation, Methodology, Writing – original draft, Writing – review & editing. **Victor Wepener:** Project administration, Resources, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Abbreviations

OCP   Organochlorine pesticides
GC-MS   gas chromatography-mass spectrometry
GC-MS/MS   gas chromatography coupled to tandem mass spectrometry
GC-ECD   gas chromatography-electron capture detector
LC-MS   liquid chromatography-mass spectrometry
LOD   limit of detection
LOQ   limit of quantification
QC/QA   Quality control and quality assurance
RSD   relative standard deviation
MDLs   method detection limits
MQLs   method quantification limits
HPLC   high-performance liquid chromatography
LC-MS   liquid chromatography mass spectrometry
QuEChERS   Quick Easy Cheap Effective Rugged and Safe
SPME   solid phase microextraction
dSPE   dispersive solid-phase extraction
LLE   liquid-liquid extraction
S/N   signal-to-noise ratio
CCS   calibration curve slope
LFB   laboratory fortified blank
GC-HRMS   gas chromatography combined with high-resolution mass spectrometry
MALD-TOF-MS   matrix-assisted laser desorption time of flight mass spectrometry
UHPLC-Q-TOF-MS   Ultra-High-performance liquid chromatography coupled with quadrupole time of flight mass spectrometry
UAE-MSC   ultrasound-assisted extraction micro scale
SPE   solid-phase extraction
SALLE   salting-out liquid-liquid extraction
DOE   Design of experiment
OFAT   One-factor-at-a-time
RSM   Response Surface Methodology

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