Review Article

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Extraction approaches for isolation of some POPs from lipid-based environmental and food matrices: A review

Supplementary material

Abbreviations

Ace	acetone
ACN	acetonitrile
ASE	accelerate solvent extraction
$C_2K_2O_4$	potassium oxalate
$CaCl_2$	calcium chloride
CH_3COOH	acetic acid
CH_3COONa	sodium acetate
COF-$B(OH)_2$	sea urchin structured COF bearing boric acid groups
Cyhex	cyclohexane
DCM	dichloromethane
DI-SPME	direct-immersion solid phase microextraction
DLLME	dispersive liquid-liquid microextraction
dSPE	dispersive solid-phase extraction
EMR-Lipid	enhanced matrix removal-lipid
Et2O	diethyl ether
EtOAc	ethyl acetate
EtOH	ethanol
GCB	graphitised carbon black
GPC	gel permeation chromatography
H_2SO_4	sulfuric acid
HBL	hydrophilic-lipophilic balanced
HCl	chloridric acid
Hex	*n*-hexane
HS-SPME	head space-solid phase micro-extraction
KOH	potassium hydroxide
LLE	liquid-liquid extraction
LPME	liquid-phase microextraction
MAE	microwave assisted extraction
MeOH	methanol
$MgSO_4$	magnesium sulphate
MOF/COF	metal organic framework/covalent organic framework
MSPD	matrix solid phase dispersion
MWCNT-MNPs	multi-walled carbon nanotube-magnetic nanoparticles
MWCNTs-SPDE	multiwalled carbon nanotubes solid phase dynamic extraction
Na_2SO_4	sodium sulphate
NaCl	sodium chloride
NaOH	sodium hydroxide
NaOH	sodium hydroxide
NDES	natural deep eutectic solvent
NH_4O_2CH	ammonium formate
NH_4OH	ammonium hydroxide
PDDA	poly(diallyl dimethilammoniumchloride)
PDMS/DVB	polydimethylsiloxane/divinylbenzene
PSA	primary secondary amine
PTFE	polytetrafluoroethylene
QuEChERS	quick, easy, cheep effective, rugged and safe
QuEChERSER	quick, easy, cheep effective, rugged, safe, efficient and robust
RP C18	reverse phase C18
SPE	solid-phase extraction
SPME	solid phase microextraction
TCM	chloroform
TEA	triethylamine

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tol	toluene
UA-MSPD	ultrasound assisted matrix solid phase dispersion
UVAE	ultrasound vortex assisted extraction

Table S1: A summary of the most used extraction methods for isolating PAHs from bovine and human milk

Classes of compounds	Sample preparation				Extraction parameters	
	Sample amount	**Pre-treatment**	**Extraction**	**Clean-up**	**Recovery (%)**	**Ref.**
PAHs	Bovine milk 250 mL	Sodium oxalate	Agitation with MeOH, Et2O, and petroleum	C18: methylene chloride and Hex (2:3)	N/A	[132]
	Bovine milk 2 g	Saponification Ethanolic solution (0.4 $mol{\cdot}L^{-1}$ NaOH)	Cyhex: Hex (1:1) -recovered with ACN after evaporation	0.45 µm PTFE filter membrane	40–130	[16]
	Human milk 2 g	Saponification Ethanolic solution (0.4 $mol{\cdot}L^{-1}$ NaOH)	Cyhex: Hex (1:1) -recovered with ACN after evaporation	0.45 µm PTFE filter membrane	90–105	[23]
	Fresh Milk 10 mL	Saponification Ethanolic solution (0.4 $mol{\cdot}L^{-1}$ NaOH)	LPME DCM: Hex (1:1); Evaporation and reconstituted into 2 mL of MeOH	N/A	85–110	[24]
	Bovine milk 2 mL	Saponification, 0.4 $mol{\cdot}L^{-1}$ NaOH with methanol/water	LLE, DCM: Hex (2:1)	C18 SPE	80–120	[25]
	Breast milk 200 µL	N/A	LLE, EtOH:H_2O_2, sonification, centrifugation	SPE PAH-column, elution using EtOAc	~99	[133]
	Bovine milk 1 g	N/A	EtOH, LLE	Semi-automated RP-C18 SPE; elution using 2-propanol	80–107	[134]
	Bovine milk 5 mL	Homogenisation with H_2O	DI-SPME for 60 min at 55°C	N/A	~100	[29]
	Commercial milk		DI-SPME for 60 min at 60°C		73–94	[30]
	Commercial milk 2 g	Saponification, ethanolic solution (1 $mol{\cdot}L^{-1}$ KOH)	Mixing with Cyhex and centrifugation, evaporation and reconstruction with ACN	Amino phase SPE and elution with Me CN	90–93	[26]
	Commercial milk 2 g	1 mL potassium Hexaferrocyanide and 1 mL zinc acetate; Saponification of the upper phase (KOH 1 $mol{\cdot}L^{-1}$)	DLLME (TCM and ACN); Centrifugation, evaporation and redissolution in 50 µL of ACN	N/A	88–100	[31]
	Commercial milk 5 g	Saponification (KOH 1 $mol{\cdot}L^{-1}$)	MeOH/ACN, sonication 7 min at 40°C Freeze lipid filtration	MWCNT-MNP + 500 mg NaCl for adsorption of PAHs; desorption using DCM, evaporate and re-dissolved in ACN/MeOH	86–100	[135]
	Commercial milk	N/A	HS-SPME with MOF/COF fiber	N/A	85–116	[136]
	Commercial milk 100 mg	Saponification (NaOH 3 $mol{\cdot}L^{-1}$) + menthol and 4 $mol{\cdot}L^{-1}$ HCl	NDES formation, Centrifugation 5 min at 5,000 rpm, 1 mL was dissolved in MeOH	0.45 µm PTFE filter membrane	70–89	[32]

Table S2: A summary of the most used extraction methods for isolating pesticides from bovine milk

Classes of compounds	Sample preparation				Extraction parameters	
	Sample amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
Pesticides	Commercial milk 10 mL	N/A	QuEChERS citrate-based buffer and ACN; ACN aliquot was filtered through a nylon filter	Oasis HBL+ nylon filtration	30–130	[41]
	Commercial milk 20 mL	N/A	QuEChERS with ACN + NaCl and $MgSO_4$, centrifugation and evaporation	1.2 mL ACN + PSA, 25 mg Z-Sep and 5 mg Z-Sep Plus adsorbent phases	35–131	[42]
	Commercial milk 20 mL	MilliQ water dilution + Carrez I and II	C18 SPE, 1.5 mL of methanol for the elution + DLLME with chlorobenzene	N/A	66–98	[43]
	Raw cow milk 5 mL	Dilution with MeOH or ACN + 1% formic acid + stirring	C18 dSPE, centrifuged and reconstituted with 1 mL MeOH	N/A	60–113	[137]
	Commercial milk 5 g	ACN extraction, centrifugation, freezing at –20°C	dSPE using PSA-Bondesil-PSA	N/A	70–120	[138]
	Goat milk 1 mL	Freezing at –20°C + 500 µL formic acid + 10 µL TEA + centrifugation	PDMS-DVB-SPME at 25°C for 55 min	N/A	81–107	[7]
	Commercial milk 1 g	ACN, shaking for 10 min and centrifuged; supernatant was filtered using 0.22 µm nylon-66 membrane	COF-$B(OH)_2$-coated fiber SPME; the fiber was placed in the vial for 40 min at 40°C	N/A	90–112	[38]
	Commercial milk 5 mL	ACN (3 mL) + NaCl, sonication and centrifugation	Supernatant fortified with 50 mg as MWCNTs- SPDE	N/A	82–112	[39]

Table S3: A summary of the most used extraction methods for isolating flame retardants from bovine and human milk

Classes of compounds	Sample preparation				Extraction parameters	
	Sample amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
FRs	Human milk 5 g	60 µL EtOAc + 5 mL of formic acid/ 2-propanol + sonication	N- vinylpyrrolidone-divinylbenzene copolymer SPE; elution MeOH/H_2O	Sulfuric acid-silica column	49–103	[49]
	Human milk 15 mL	360 µL formic acid + 15 mL ACN + agitation	Supernatant collected and evaporated; reconstitution using 0.5 mL of MeOH	N/A	87–108	[47]
	Human milk 1 g	5 mL 0.5% formic acid in ACN + agitation, ultrasonication, centrifugation	d-SPE using $MgSO_4$ + PSA + C18; centrifugation, evaporation and reconstitution using H_2O/MeOH	N/A	12–97	[8]
	Human milk 0.5 g	5 mL 0.5% formic acid in ACN, agitation, ultrasonication, centrifugation	QuEChERS, $MgSO_4$ + PSA + C18; centrifugation, evaporation and reconstitution using H_2O/MeOH	SPE, Supernatant was purified using ProElut PLS extraction cartridge; elution using ACN; evaporation to dryness and reconstitution using MeOH	75–125	[14]
	Human milk 30 mL	Fortification Na_2SO_4	ASE, 2 cycles at 110°C using Ace/Hex	GPC using EtOAc/Cyhex, evaporation and reconstitution using Hex+ 98% H_2SO_4	79–109	[50]
	Human milk 5 g	1 mL $C_2K_2O_4$ (2%) + 10 mL EtOH + 5 mL Et_2O + mixing	Extraction with 10 mL of pentane; evaporation and reconstitution with DCM	Filtration using 0.45 µm + GPC	~60	[48]
	Human milk 4 g	Freeze-drying + homogenisation	ASE, Hex: Ace three cycles, concentration to 1 mL	Multilayer silica (neutral silica, acid silica and Na_2SO_4) column, elution with Hex: DCM	63–135	[51]
	Cow milk 0.5 g	5 mL 0.5% formic acid in ACN, agitation, ultrasonication, centrifugation	QuEChERS $MgSO_4$ + PSA + C18; centrifugation, evaporation and reconstitution using H_2O/MeOH	SPE, Supernatant was purified using ProElut PLS extraction cartridge; elution using ACN; evaporation to dryness and reconstitution using MeOH	75–125	[14]
	Cow milk 10 mL	Acidification using 1 mL HCl + sonication	Homogenisation using Ace/Hex + centrifugation (all procedure was repeated twice); evaporation and reconstitution using DCM;	GPC using DCM/Hex + Florisil SPE and eluted using DCM: Hex	30–92	[139]

Table S4: A summary of the most used extraction methods for isolating polychlorobiphenyls (PCBs) from bovine and human milk

Classes of compounds	Sample preparation				Extraction parameters	
	Sample amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
PCBs	Human milk N/A	Addition of EtOH	125 mL Hex/Ace + Mixing; Evaporation up to 2 mL	Multilayer Silica gel column (Na_2SO_4; deactivated silica; acidic silica); elution using Hex/DCM	79–93	[19]
	Commercial milk 95 g	Sodium oxalate (1 g) + 20 mL anhydrous alcohol + acid hydrolysis (H_2SO_4, 2 M) + filtration	Automated Soxhlet extraction using Hex (100 mL)	GPC + acid/neutral silica mini-column and collected with Hex	99–134	[1]
	Commercial milk 5 mL	5 mL of 50% w/v NaOH and 1 mL of Ace	SPE combined with DLLME using Hex and Florisil column; Elution with Hex; DLLME using chlorobenzene (19 µL)	N/A	66–103	[105]
	Commercial milk 11 g	Hex/Ace (100 mL) + agitation + sonication	Extraction using agitation + sonication; Evaporation of supernatant to 4 mL	H_2SO_4 (98%) treatment overnight; centrifugation and evaporation of supernatant; Residues recovered using Hex and loaded into a Bond Elut-PCB cartridge; Elution with Hex	15–120	[140]
	Commercial milk N/A	Saponification with NaOH	HS-SPME using PDMS at 100°C	N/A	75–102	[53]
	Commercial milk 5 mL	Saponification with $NH_4(OH)$; Et2O + Pet Ether + centrifugation	HS-SPME using PDMS at 96°C for 60 min	N/A	N/A	[141]
	Commercial milk 5 mL	Addition of MeOH (400 µL) + addition of MilliQ for 24 h, at 4°C + Addition of NaCl + agitation	HS-SPME using PDMS at 65°C for 45 min	N/A	94–122	[54]
	Human milk 5 mL	Addition of H_2O + ACN saturated in Hex + mixing; Addition of salts ($MgSO_4$, NaCl; sodium citrate monobasic; $Na_2C_6H_6O_7$); Mixing of the solution;	QuEChERS extraction ACN/Hex (5 mL)	Adding $MgSO_4$ + PSA; Evaporation and residues dissolved in Hex and H_2SO_4	96–120	[142]

(Continued)

Table S4: *Continued*

Classes of compounds	Sample preparation				Extraction parameters	
	Sample amount	**Pre-treatment**	**Extraction**	**Clean-up**	**Recovery (%)**	**Ref.**
	Human milk 50 mL	Addition of $C_2K_2O_4$ + EtOH + ether and pentane + evaporation and fat isolation	2 g of milk fat were clean on a silica gel column and eluted using Hex	Chromosorb WHP/Charcoal SP-1 column	N/A	[143]
	Cow milk 250 mL	Addition of detergent solution + agitation at 90°C; fat was recovered and filtered	SPE of 0.5 g of milk fat using Florisil and anhydrous Na_2SO_4; elution using Hex/DCM; evaporation and reconstitution using 2 mL Hex/DCM; evaporation and reconstitution using 500 µL of Hex	N/A	N/A	[144]

Table S5: A summary of the most used extraction methods for isolating PAHs from meat

Classes of compounds	Sample preparation				Extraction parameters	
	Sample amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
PAHs	Sausages 3 g	Mincing	QuEChERS, ACN: Ace (3:2)	EMR Lipid Tube, polish tube, evaporation and recovered with DCM	64–119	[13]
	Ground pork 2 g	N/A	QuEChERS, ACN+ 1% CH_3COOH and MgSO4 and CH3COONa	PSA, MgSO4 and C18, evaporation and reconstitution with Hex	80–101	[18]
	Pork/Beef 10 g	N/A	QuEChERS, H_2O, ACN homogenisation and addition of $MgSO_4$, NaCl, sodium citrate dihydrate, sodium hydrogencitrate sesquihydrate, centrifugation	$MgSO_4$, NaCl, centrifugation, evaporation and reconstitution with ACN	69–73	[9]
	Pork/Beef 10 g	Saponification Ethanolic solution (1 mol·L^{-1} KOH)	Cyhex, centrifugation, evaporation	C18 SPE column (Cyhex, ACN), evaporation, reconstitution with Hex, Florisil SPE column (DCM, Hex), evaporation to dryness and reconstitution with ACN	86–90	[9]
PAHs	Pork/Beef 2 g	Lyophilization	Hex, sonication, filtration, evaporation, redissolution in Hex	Silica cartridge, elution with Hex: DCM (70:30), evaporation, redissolution with ACN	74–82	[9]
	Beef 5 g	Homogenisation, immersion in a buffer solution, lyophilization	Hex, ACN, sonication, centrifugation, evaporation, reconstitution with Hex.	Florisil SPE cartridge elution using Hex: DCM (70:30), evaporation, redissolution with ACN	82–102	[145]
	Lamb 2 g	N/A	QuEChERSER; ACN/H_2O (4:1), agitation, centrifugation, addition of MgSO4/NaCl (4:1)	Supernatant cleaned up by μSPE ($MgSO_4$, C18, PSA, GCB)	24–140	[17]
	Beef 2 g	Homogenisation	ASE with ACN: EtOAc (1:3), evaporation, redissolution in Cyhex	Dual layer SPE cartridge (Florisil, mix C18-Silica gel), elution using ACN/ EtOAc (97:3), evaporation until 0.5 mL	72–109	[12]
	Pork 5 g	Mincing	H_2O and ACN vortexing and addition of $MgSO_4$, NaCl, shacking, centrifugation, evaporation to dryness and redissolution in 100 μL of ACN	N/A	94–109	[146]
PAHs	Kebabs 5 g	Freezing and homogenisation	KOH (1mol·L^{-1}), MeOH-ACN (30%), sonication, centrifugation, freeze lipid filtration	Addition of MWCNT-MNP composite and NaCl, vortexing. Addition of DCM to elute the analytes. Evaporation to dryness and reconstitution with MeOH-ACN (50:50)	81–97	[59]
	Sheep heart 5 g	Homogenisation	QuEChERS; EtOAc, Ace, Isooctane (2:2:1), H_2O, shaking, NaCl, NH_4O_2CH, agitation, centrifugation, separation of the supernatant	Z-Sep, PSA, centrifugation	73–111	[57]

Table S6: A summary of the most used extraction methods for isolating pesticides from meat

Classes of compounds	Sample preparation				Extraction parameters	
	Sample amount	**Pre-treatment**	**Extraction**	**Clean-up**	**Recovery (%)**	**Ref.**
Pesticides	Chicken meat and liver 5 g	Lyophilization, homogenisation, incubation with β-Glucoronidase	Pre-extraction with ACN, MgSO4, NaCl followed by NADES-based DLLME extraction with Thymol/ Menthol (1:1)	N/A	81–112	[63]
	Pork lard, Mutton tallow 5 g	Melting	LLE; H_2O, ACN, agitation, $MgSO_4$, NaCl	Low temperature assistant clean up: immersion in liquid nitrogen 20 min Collection of supernatant and clean up by d-SPE with Allumina and $MgSO_4$	70–120	[19]
	Lamb 2 g	N/A	QuEChERSER; ACN/H_2O (4:1), agitation, centrifugation, addition of MgSO4/NaCl (4:1)	Supernatant cleaned up by μSPE ($MgSO_4$, C18, PSA, GCB)	2–112	[17]
	Pancetta, Tenderloin, Budiola, Sausage 3 g	N/A	QuEChERS; ACN, $MgSO_4$, CH_3COONa, agitation, centrifugation, separation of upper phase (ACN)	$MgSO_4$, PSA, C18, centrifugation, evaporation, reconstitution with Hex	82–116	[62]
	Pork, beef, ham, sausage, liverpaté, reindeer, chicken N/A	Homogenisation	Liquid/liquid lipid extraction, Cyhex:Ace (3:2), NaCl, H_2O, homogenisation, sonication, centrifugation, evaporation and reconstitution with Cyhex	Lipid fraction remotion with H_2SO_4, evaporation to a final volume of 0.2–0.3 mL	66–126	[10]

Table S7: A summary of the most used extraction methods for isolating PCB from meat

Classes of compounds	Sample preparation				Extraction parameters	
	Sample amount	**Pre-treatment**	**Extraction**	**Clean-up**	**Recovery (%)**	**Ref.**
PCB	Chicken, chicken liver, goat 5 g	Homogenisation with liquid nitrogen. Centrifugation after the addition of NaCl and MgSO4.	DLLME using DES (thymol-canphor, 1:1) and H_2O, agitation and centrifugation. Collection of the upper phase (DES), dilution with ACN.	N/A	81–95	[11]
	Sausages 5 g	Homogenisation	ACN and $MgSO_4$, agitation, centrifugation, collection of the supernatant.	Fe3O4@SiO2@PSS/ PDDA/MWCNTs nanoparticles, agitation, collection of the supernatant, evaporation. Elution of the magnetic adsorbent with DCM Hex (60:40), evaporation. Dissolution of the residues with Hex.	93–103	[65]
	Sheep heart 5 g	Homogenisation	QuEChERS; EtOAc, Ace, Isooctane (2:2:1), H_2O, shaking, NaCl, NH_4O_2CH, agitation, centrifugation, separation of the supernatant	Z-Sep, PSA, centrifugation	81–116	[57]
PCB	Lamb 2 g	N/A	QuEChERSER; ACN/H_2O (4:1), agitation, centrifugation, addition of MgSO4/NaCl (4:1)	Supernatant cleaned up by µSPE ($MgSO_4$, C18, PSA, GCB)	41–99	[17]
	Pancetta, tenderloin, budiola, sausage 3 g	N/A	ACN, $MgSO_4$, CH_3COONa, agitation, centrifugation, separation of upper phase (ACN)	$MgSO_4$, PSA, C18, centrifugation, evaporation, reconstitution with Hex	100–112	[62]
	Bovine adrenal fat, sausage, salami, soudjouk 1 g	Melting	QuEChERS: Ace, agitation, centrifugation, evaporation to dryness and resuspension in ACN.	$MgSO_4$, PSA, agitation, centrifugation, evaporation and resuspension in isooctane	96–102	[64]
	Pork, beef, ham, sausage, liverpaté, reindeer, chicken N/A	Homogenisation	Liquid/liquid lipid extraction, Cyhex:Ace (3:2), NaCl, H_2O, homogenisation, sonication, centrifugation, evaporation and reconstitution with Cyhex	Lipid fraction remotion with H_2SO_4, evaporation to a final volume of 0.2–0.3 mL	66–126	[10]

Table S8: A summary of the most used extraction methods for isolating FRs from meat

Analytes	Samples amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
BFRs	Chicken breast 2 g	Homogenised and lyophilised	Ultrasonic extraction with 5 ml of ACN: tol (9:1) for 10 min.	Florisil cartridges eluted with 6 mL of Hex: DCM (4:1) followed by 6 mL of Hex: DCM (1:1). Further clean-up with an acidified silica eluted with 12 mL od Hex: DCM (1:1)	75–121	[66]
	Beef, pork, lamb, game, poultry, cured and processed meat 0.5 g	Lyophilised	5 ml of ACN was added to sample overnight. The solvent was concentrated, and QuE Z-Sep sorbent power was added to a d-SPE. The extract was concentrated and reconstituted in Hex	Florisil cartridges	53–71	[73]
	Pork, beef and chicken meat	Grinded	ASE with Hex: Ace (1:1)	Column packed with acidified silica and Na_2SO_4 eluted with Hex: DCM	88–106	[67]
	Processed meats, poultry and carcase meats 4 g	Homogenised and lyophilised	Liquid-solid extraction with Hex: DCM	Adsorption chromatography on alumina	50–110	[70]
	Meat and meat products 5–30 g	Lyophilised and ground with Na_2SO_4	Soxhlet extraction with Hex: Ace (1:1) for 20 h. The extract was evaporated to dryness and redissolved in EtOAc Cyhex (1:1)	GPC eluted with EtOAc: Cyhex (1:1). The extracts were evaporated and reconstituted in Hex and followed by sulfuric acid treatment.	70–130	[50]
	Chicken, beef and mutton	Washed, homogenised and mixed with Na_2SO_4	Soxhlet extraction with Hex: Ace (1:1) for 24 h.	Column packed with activated silica, silica treated with NaOH, silica treated with H_2SO_4 and granular Na_2SO_4. Eluted with Hex: DCM (97:3)	99–110	[69]
	Meat 2.5 g	Homogenised and lyophilised	Solid-liquid extraction with Hex: DCM (1:1)	SPE cartridge packed with acidified silica gel and eluted with DCM Then the solvent was exchanged to ACN	70–120	[71]
	Lean beef, lean pork, bacon and chicken thigh 2.5 g	Homogenised and lyophilised	UVAE for 1 h with ACN: tol mixture (9:1)	Florisil cartridges and fractionation. Further clean-up differs between the fraction	66–135	[72]

Table S9: A summary of the most used extraction methods for isolating pesticidi from olive oil

Classes of compounds	Sample preparation				Recovery (%)	Ref.
	Sample amount	Pre-treatment	Extraction	Clean-up		
Pesticides	5 g	Dissolution in 5 mL of Hex	LLE with 10 ml of ACN solvent	Envi-Carb SPE cartridge, Diol SPE cartridge	71–109	[147]
	2 g	N/A	Liquid partitioning in Hex (2 mL) and ACN (10 mL) supplied with 3 mg of anhydrous sodium sulphate	GPC clean-up columns packed with styrene-divinylbenzene copolymer with DCM as mobile phase	84–110	[76]
	5 g	Dissolution in 5 mL of Hex	SPE with 10 mL of CAN, SPE with 12 mL of ACN/tol (95:5, v/v)	ENVI-Carb SPE cartridge, Diol SPE cartridge	96–105 (GC-NPD method), 89–102 (GC-ECD method)	[82]
	5 g	Liquid/liquid partitioning with 15 mL of petroleum ether saturated with 25 mL of ACN	MSPD using aminopropyl as sorbent material and ACN as eluting solvent	Florisil sorbent	85–115	[77]
	0.5 g	dissolution and homogenization in tetrahydrofuran (THF)	Extraction by size-exclusion chromatography	N/A	3–99 (GC-MS analysis), 28–92 (HPLC-MS analysis)	[148]
	15 g	Mixing with 25 mL Hex	SPE with 20 mL ACN saturated with Hex	Extrelut-QE mini-column packed with C18 and alumina phases	74–105	[93]
	5 g	N/A	MAE with 5 mL of ACN–DCM (90:10, v/v) extracting solution	Envi-Carb SPE cartridge	62–99	[149]
	3 g	Mixing with 7g of water, Liquid/liquid partitioning with 25 mL of ACN saturated with petroleum ether	Liquid/liquid partitioning with 10 mL of ACN, 4 g of anhydrous magnesium sulphate and 1 g of NaCl (modified QuEChERS method), MSPD with aminopropyl sorbent	d-SPE clean-up with graphitized carbon black, primary-secondary amine and C18 sorbents, Florisil SPE cartridge	70–130, 21–160	[78]
	5 g	Homogenization and dissolution in 5 mL of Hex	Two steps of LLE with 5 mL of ACN for each step followed by UA-MSPD	PSA as dispersant sorbent and 2 g of Florisil/GBC (70:30 w/w) as clean-up sorbent	74–104	[150]

(Continued)

Table S9: *Continued*

Classes of compounds	Sample preparation				Recovery (%)	Ref.
	Sample amount	Pre-treatment	Extraction	Clean-up		
	10 g	N/A	Liquid partitioning with MeOH (10 mL) with 1% of formic acid and 10 mL of ultrapure water	N/A	70–120	[151]
	3 g	N/A	QuEChERS	EMR-Lipid sorbent	75–119	[152]
	0.50 g	Vortexing with 4 mL of Hex	Vortex-assisted emulsification liquid-liquid microextraction with 200 µL of deep eutectic solvent	Hex/ACN/DES, solvent system and hydrophilic [chloride: urea] DES	63–119	[153]
	5 g	N/A	QuEChERS with 10 mL of ACN	GPC clean-up column rinsed with 30 mL of Hexane/DCM (1:1, v:v)	N/A	[154]
	3 g	Mixing with 7 mL of water	QuEChERS with 10 mL of ACN	EMR-Lipid and Z-Sep+/PSA/$MgSO_4$ sorbent	70–113, 72–107	[84]
	3 g	N/A	QuEChERS with 10 mL of ACN	$MgSO_4$/PSA/C18	80–95	[155]

Table S10: A summary of the most used extraction methods for isolating PAHs from olive oil

Classes of compounds	Sample preparation				Recovery (%)	Ref.
	Sample amount	Pre-treatment	Extraction	Clean-up		
PAHs	1 g	GPC	GPC	GPC	52–110	[156]
	2.5 g	Dissolution in 10 mL of Hex	SPE with Hex and DCM mixture (70:30, v/v)	Silica SPE cartridge	32–152	[88]
	2.5 g	Dissolution in 10 mL of Hex	SPE with Hex and DCM mixture (70:30, v/v)	Silica SPE cartridge	N/A	[157]
	0,5 g	N/A	Extraction with 3 mL of ACN followed by the extraction of 2.5 mL of the ACN extract in 4 mL of *Hex*	DI-SPME with carbopack Z/DPMS fiber	N/A	[158]
	5 g	N/A	QuEChERS with 10 mL of ACN	GPC clean-up column rinsed with 30 mL of Hexane/DCM (1:1, v:v)	N/A	[154]
	0.4 g	N/A	SPE with 15 mL of ACN	Supelclean EZ-POP NP cartridge	85–98	[85]
	5 g	Mixing and equilibration with 10 mL of ACN/Ace (6:4)	LLE with ACN/Ace (6:4, v/v)	EZ-POP NP dual-layer and NH_2 SPE cartridge	98–102	[87]
	0.4 g	N/A	SPE with 15 mL of ACN	Dual-layer cartridge containing activated Florisil and a mixture of octadecyl (C18)-bonded and zirconia-coated silicas sorbents	83–127	[159]
	2.5 g	N/A	LLE with 10 mL of ACN/Ace (60:40, v/v) mixture	Sep-Pak C18 and Sep-Pak Florisil SPE cartridges	N/A	[160]
	2 g	N/A	LLE with 10 mL of ACN-Ace (6:4, v/v) mixture	SPE C18 cartridge	70–120	[86]
	2.5 g	N/A	LLE with 10 mL of ACN/Ace (60:40, v/v) mixture	C18 and Florisil SPE cartridges	30–121	[94]
	5 g	N/A	HS technique	N/A	96–99	[161]
	2 g	N/A	HS-SPME	Divinylbenzene/carboxen/ polydimethylsiloxane fiber	74–77	[92]
	1 g	N/A	LLE with 10 mL of ACN	GPC	75–115	[91]

Table S11: A summary of the most used extraction methods for isolating PCBs from olive oil

Classes of compounds	Sample preparation				Recovery (%)	Ref.
	Sample amount	Pre-treatment	Extraction	Clean-up		
PCBs	5 g	N/A	QuEChERS with 10 mL of ACN	GPC clean-up column rinsed with 30 mL of Hexane/DCM (1:1, v: v)	N/A	[154]
	15 g	Mixing with 25 mL of Hex	SPE with 20 ml of ACN saturated with Hex	Extrelut-QE mini-column with C18 and alumina phases	60–119	[93]
	5 g	N/A	Digestion with KOH/ethanol solution	Silica gel column	90–105	[162]
	5 g	N/A	Cold extraction with 20 ml of Hex	Purification with 5 mL of H_2SO_4 (96%)	60–74	[163]
	10 g	Dissolution in 50 mL of DCM	Extraction onto a Carbosphere column with 40 mL of tol	Column with 0.5 g of 44% H_2SO_4-silicagel and 5 g of alumina phases	N/A	[94]
	0.5 g	Dissolution in 5 mL of Hex	Extraction onto a column with 10 g 44% H_2SO_4-silicagel with 100 mL of Hex	Column with alumina		
	0.5–3.0 g	Homogenization and mixing with Na_2SO_4	Extraction with Hex	Acidified silica gel column (H_2SO_4, 44% w/w) elution with 50 mL of Hex-DCM mixture (1/1, v/v)	85–120	[95]
	5 g	N/A	Digestion with KOH/ethanol solution	Multilayer silica gel column	90–105	[96]

Table S12: A summary of the most used extraction methods for isolating FRs from olive oil

Classes of compounds	Sample preparation				Recovery (%)	Ref.
	Sample amount	Pre-treatment	Extraction	Clean-up		
FRs	0.20 g	Homogenization and freeze-drying	Vortex and ultrasound extraction with 5 mL of ACN: tol (9:1, v/v)	First clean-up with Florisil cartridge followed by a further clean-up through 1.5 g of acidified silica (AS, 5%, w/w)	75–125	[66]
	2.5 g	Shaking with 15 mL of ACN: formic acid (9:1)	Extraction with acidified ACN (QuEChERS)	dSPE with EMR-Lipid sorbent and acidified silica (44%, w/w)	70–120	[71]

Table S13: A summary of the most used extraction methods for isolating PAHs from fish

Analytes	Sample amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
PAHs	*Sarotherodon melanotheron* and *Clarias gariepinus* 1 g	Homogenised	Two rounds of ultrasonic extraction with methanol, and two rounds with DCM and then methanol: DCM (1:1)	Chromatographic column filled with activated alumina and silica gel (1:3) and eluted with DCM: Hex (3:2)	N/A	[102]
	Cynoscion guatucupa, *Micropogonias furnieri*, *Mustelus schmitti* and *Ramnogaster arcuata* 3–5 g	Homogenised, lyophilised and digested under reflux with methanol for 8 h. Then, KOH (0.7 M) and tridistilled water were added and refluxed for 2 h	The non-saponifiable fraction was extracted with Hex	Alumina-silica (2:1) gel column eluted with Hex and Hex: DCM (7:3) mixture	75–105	[99]
	Fatty fish meat 5 g	Homogenised	The sample was mixed with 5 mL od distilled water, 15 mL of ACN + 1% acetic acid and 2 mL of Hex and vortexed. Then 6 g of $MgSO_4$ and 1.5 g of CH_3COONa were added and centrifugated. 1.5 mL of the ACN portion was pipetted out	dSPE with 100 mg of $CaCl_2$ and 150 mg $MgSO_4$ Supernatant was further cleaned with 50 mg of PSA, 50 mg of Florisil, 150 mg C18 and 150 mg of $MgSO_4$ and centrifugated The supernatants were filtered through PTFE membrane	60–120	[97]
	Turbot and salmon 1 g	Lyophilised then reconstituted	MAE after the addition of saturated MeOH-KOH and Hex	SPE with Florisil	~90	[100]
	Blue mussels (*Mytilus edulis*) and Atlantic salmon (*Salmo salar*) 6 g	Homogenised and mixed with 5 mL of distilled H_2O	QuEChERS extraction with EtOAc, NaCl, Na_2SO_4	GPC eluted with DCM and recovered with Hex and then SPE column filled with silica and Na_2SO_4 and eluted with Hex: DCM (3:1).	~100	[101]

Table S14: A summary of the most used extraction methods for isolating PCBs from fish

Analytes	Samples amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
PCBs	Dorsal muscle of *Anguilla anguilla* 0.5 g	Lyophilised and homogenised	Soxhlet extraction with 50 mL of Ace: Hex (1:1). After the evaporation of the solvents, lipids were suspended in 2 mL of Hex	Lipids were destroyed with 5 mL of H_2SO_4 The extracts were concentrated and cleaned-up on a Florisil column	91–102	[104]
	Blue mussels (*Mytilus edulis*) and Atlantic salmon (*Salmo salar*) 6 g	Homogenised and mixed with 5 mL of distilled H_2O	QuEChERS extraction with EtOAc, NaCl, Na_2SO_4	GPC eluted with DCM and recovered with Hex and then SPE column filled with silica and Na_2SO_4 and eluted with Hex: DCM (3:1).	~100	[101]
	Marine fish 2 g	Lyophilised, homogenised and mixed with anhydrous Na_2SO_4	Soxhlet extraction with Hex: DCM mixture (1:3)	GPC column and multilayer silica gel column packed with Florisil, anhydrous Na_2SO_4 and neutral and acidified silica	81–106	[105]
	Tilapia and salmon	Homogenised	QuEChERS with ACN	Syringes equipped with a polyethylene frit stored at −24°C for 2 h. Then the sample was shacked and centrifugated first with $CaCl_2$ and then with $MgSO_4$.	70–115	[108]
	Thunnus thynnus, Pagellus bogaraveo Dentex dentex 2 g	Homogenised with diatomaceous earth and Na_2SO_4	ASE with Hex: Ace (4:1) mixture. The cells were packed with cellulose filter and fat retainer (neutral silica).	N/A	90–105	[164]
	Notothenia rossii and *Trematomus newnesi*	Lyophilised and homogenised	Soxhlet extraction with Hex: Ace (3:1)	Acidified silica column eluted with Hex and DCM. The eluent was dried and reconstituted with isooctane	>92	[103]

Table S15: A summary of the most used extraction methods for isolating FRs from fish

Analytes	Leaves samples	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
BFRs	Blue mussels (*Mytilus edulis*) and Atlantic salmon (*Salmo salar*) 6 g	Homogenised and mixed with 5 mL of distilled water	QuEChERS extraction with EtOAc, NaCl, Na_2SO_4	GPC eluted with DCM and recovered with Hex and then SPE column filled with silica and sodium sulphate and eluted with Hex: DCM (3:1).	~100	[101]
	Marine fish 2 g	Lyophilised, homogenised and mixed with anhydrous Na_2SO_4	Soxhlet extraction with Hex: DCM (1:3)	GPC column and multilayer silica gel column packed with Florisil, anhydrous Na_2SO_4 and neutral and acidified silica	82–110	[105]
	Notothenia rossii and *Trematomus newnesi*	Lyophilised and homogenised with Na_2SO_4	Soxhlet extraction with Hex: Ace (3:1)	Acidified silica column eluted with Hex and DCM. The eluent was dried and reconstituted with isooctane	>92	[103]

Table S16: A summary of the most used extraction methods for isolating pesticides from fish

Analytes	Samples amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
Pesticides	Dorsal muscle of *Anguilla anguilla* 0.5 g	Lyophilised and homogenised	Soxhlet extraction with 50 mL of Ace: Hex (1:1). After the evaporation of the solvents, lipids were suspended in 2 mL of Hex	Lipids were destroyed with 5 mL of H_2SO_4. The extracts were concentrated and cleaned-up on a Florisil column	90–108	[104]
	Fatty fish meat 5 g	Homogenised and mixed with 5 mL of distilled H_2O	15 mL of ACN + 1% CH_3COOH and 2 mL of Hex were added to the samples and vortexed. Then 6 g of $MgSO_4$ and 1.5 g of CH_3COONa were added and centrifugated. 1.5 mL of the ACN portion was pipetted out	dSPE with 100 mg of $CaCl_2$ and 150 mg $MgSO_4$. Supernatant was further cleaned with 50 mg of PSA, 50 mg of Florisil, 150 mg C18 and 150 mg of $MgSO_4$ and centrifugated. The supernatants were filtered through PTFE membrane.	60–120	[97]
	Marine fish 2 g	Lyophilised, homogenised and mixed with anhydrous Na_2SO_4	Soxhlet extraction with Hex: DCM mixture (1:3)	GPC column and multilayer silica gel column packed with Florisil, anhydrous Na_2SO_4 and neutral and acidified silica	83–112	[105]
	Tilapia and salmon	Homogenised	QuEChERS with ACN	Syringes equipped with a polyethylene frit stored at −24°C for 2 h. Then the sample was shacked and centrifugated first with $CaCl_2$ and then with $MgSO_4$.	70–115	[108]
	Thunnus thynnus, Pagellus bogaraveo Dentex dentex 2 g	Homogenised with diatomaceous earth and Na_2SO_4	ASE with Hex: Ace (4:1) mixture. The cells were packed with cellulose filter and fat retainer (neutral silica).	N/A	78–97	[164]
	Notothenia rossii and *Trematomus newnesi*	Lyophilised and homogenised with Na_2SO_4	Soxhlet extraction with Hex: Ace (3:1)	Acidified silica column eluted with Hex and DCM. The eluent was dried and reconstituted with isooctane	>92	[103]

Table S17: A summary of the most used extraction methods for isolating PAHs from leaves

Analytes	Samples amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
PAHs	Pine needles 5 g	Minced	ASE with ACN	SPE with a CHROMABOND® EASY. Elution with 2 mL of EtOAc, tol and ACN	83–100	[126]
	Sambucus nigra and *Acacia melanoxylon* 2 g	Lyophilized	Ultrasonic bath for 10 min with 20 mL of DCM: Hex mixture (1:1) for 3 times	SPE with a Seo-Pak alumina cartridge. Elution with 10 mL of DCM: Hex mixture and 5 mL of DCM	48–106	[120]
	Pine needles (*P. mugo*)	Minced and lyophilized	Automatic extractor using DCM	Activated silica gel and GPC	72–102	[110]
	Quercus ilex 5 g	N/A	Ultrasonic bath for 3 min in pulse mode with 100 mL DCM: Ace mixture (1:1) for 3 times	Filtration through PTFE filters (0.2 µm)	40–80	[122]
	Photinia, Mahonia, Loropetalum, Hypericum, Rhododendron, Pinus	Minced and lyophilized	Ultrasonic bath for 30 min with Hex: Ace (1:1) for 3 times	Silica gel column eluted with Hex: DCM (1:1) and syringe filter (0.2 µm)	45–119	[112]
	Picea spinulosa, *Abies spectabilis* and *Pinus densata* 1 g	Lyophilized and minced	ASE with 70 mL DCM: Hex mixture (1:1)	Glass column filled with anhydrous Na_2SO_4, de-activated alumina and de-activated silica gel. DCM: Hex mixture (2:3) was used to elute and further cleaned-up by GPC	50–120	[113]
	Murraya paniculate 10 g	Minced and lyophilized	Soxhlet extracted for 6 h in 120 mL of tol	Silica gel column chromatography filled with anhydrous Na_2SO_4 (2 g) and preheated silica gel (10 g) eluted with 25 mL of Hex	N/A	[114]
	Pine needles 5–10 g	N/A	Storage of the samples with 40 mL of Ace overnight and ultrasonic bath for 30 min	GPC filled with silicic acid (3 g) and alumina (2 g) and elution with EtOAc: Cyhex (1:1)	52–111	[127]
	Olive tree 5 g	N/A	Addition of 50 mL of DCM: petroleum ether (1:1) and shacking for 24 h, then ultrasonic bath for 15 min	Column filled with silicic, alumina and Na_2SO_4. Elution with DCM	50–120	[130]
	Sambuca nigra 2 g	Lyophilized	Ultrasonic bath for 10 min with 20 mL of DCM: Hex (1:1) for 3 times	SEP-Pak Alumina cartridges. Elution with DCM.	65–106	[121]
	Abies holophylla and *Pinus tabuliformis* 0.5 g	Minced	Ultrasonic bath for 15 min with 10 mL of DCM for 3 times.	SPE column and eluted DCM: Hex (1:1)	64–120	[56]

(Continued)

Table S17: *Continued*

Analytes	Samples amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
	Berberis thunbergia, *Sabina Chinensis*, *Euonymus japonicas*, *Juniperus sabina*, *Buxus microphylla*, *Pinus tabuliformis* and *Pinus bungeana* 5 g	Minced	Ultrasonic bath extraction for 30 min with 50 mL of DCM: Hex (1:1)	SPE column of 800 mg silica gel and 1200 mg neutral alumina.	71–98	[117]
	Ligustrum lucidum 2.5 g	N/A	Ultrasonic bath extraction for 30 min with 100 mL of DCM: Hex (1:1)	SPE in glass column packed with 5 g of aluminium oxide and topped with Na_2SO_4. Eluted with 50 mL DCM: Hex. GPC with glass column filled with 6 g of S-X3 Bio-beads in DCM: Hex. Eluted with 40 mL of DCM: Hex.	99	[129]
	Citrus aurantium 1 g	Minced	Sonication for 10 min with 3 mL of Ace	d-SPE with 0.5 g of C18 sorbent	55–102	[118]

Table S18: A summary of the most used extraction methods for isolating PCBs from leaves

Analytes	Leaves samples	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
PCBs	Pine needles 10 g	Minced	ASE with ACN	SPE with a CHROMABOND® EASY. Elution with 2 mL of EtOAc, tol and ACN	76–107	[165]
	Pine needles 5 g	Minced	ASE with ACN	SPE with a CHROMABOND® EASY. Elution with 2 mL of EtOAc, tol and ACN	80–99	[126]
	Pine needles (*P. mugo*)	Minced and lyophilized	Automatic extractor using DCM	Activated silica gel modified with H_2SO_4	88–100	[110]
	Ligustrum lucidum 2.5 g	Minced	Ultrasonic bath for 30 min with DCM: Hex (1:1)	Alumina SPE glass column and GPC	87	[111]
	Pine needles 5–10 g	N/A	Storage of the samples with 40 mL of Ace overnight and ultrasonic bath for 30 min	GPC filled with silicic acid (3 g) and alumina (2 g) and elution with EtOAc: Cyhex (1:1)	58–108	[127]
	Olive tree (*Olea Europaea*) leaves 5 g	Minced	Addition of 50 mL of DCM: Petroleum ether (1:1) and shacking for 24 h, then ultrasonic bath for 15 min	Column filled with silicic acid, alumina, and sodium sulphate. The extracts were eluted with PE. Before the injection a GPC column, filled with biobead S-X3, was applied using Hex: DCM mixture as solvent.	52–90	[131]
	Lolium multiflorum	Homogenized and lyophilized	Soxhlet extraction with tol for 24 h.	Multi-layer column with acid and basic silica gel and alumina column	N/A	[119]
	Pine needls 3 g	N/A	Supercritical fluid extraction using pure CO_2 at 2.5 mL/min, 120°C for 50 min and then collected using 3 mL Hex	N/A	>90	[128]

Table S19: A summary of the most used extraction methods for isolating pesticides from leaves

Analytes	Samples amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
Pesticide	Pine needles 10 g	Minced	ASE with ACN	SPE with a CHROMABOND® EASY. Elution with 2 mL of EtOAc, tol and ACN	76–107	[165]
	Pine needles 5 g	Minced	ASE with ACN	SPE with a CHROMABOND® EASY. Elution with 2 mL of EtOAc, tol and ACN	80–100	[126]
	Pine needles (*P. mugo*)	Minced and lyophilized	Automatic extractor using DCM	Activated silica gel modified with H_2SO_4	75–98	[110]
	Ligustrum lucidum 2.5 g	Minced	Ultrasonic bath for 30-min with DCM: Hex (1:1)	Alumina SPE glass column and GPC	74	[111]
	Picea spinulosa, *Abies spectabilis* and *Pinus densata* 1 g	Lyophilized and minced	ASE with 70 mL DCM: Hex (1:1)	Acid silica gel/Florisil glass column and eluted using 50 mL of DCM: Hex (2:3). and further cleaned-up by GPC	52–117	[113]

Table S20: A summary of the most used extraction methods for isolating FRs from leaves

Analytes	Samples amount	Pre-treatment	Extraction	Clean-up	Recovery (%)	Ref.
BFRs	Pine needles (*P. mugo*)	Minced and lyophilized	Automatic extractor using DCM	Activated silica gel modified with H_2SO_4	50–80	[110]
	Ligustrum lucidum 2.5 g	Minced	Ultrasonic bath for 30-min with Hex: DCM (1:1)	Alumina SPE glass column and GPC	84	[111]
	Pine needles 5–10 g	N/A	Storage of the samples with 40 mL of Ace overnight and ultrasonic bath for 30 min	GPC filled with silicic acid (3 g) and alumina (2 g) and eluation with EA: Cyhex (1:1)	72–113	[127]
	Bitter orange (*Citrus aurantium)* 0.5 g	Minced	Sonication for 15 min at 30°C with 6 mL of Ace: ACN (2:1)	d-SPE with 0.28 g of Florisil sorbent for 2 min	63	[109]

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