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Identification of hazardous organic compounds in e-waste plastic using non-target and suspect screening approaches

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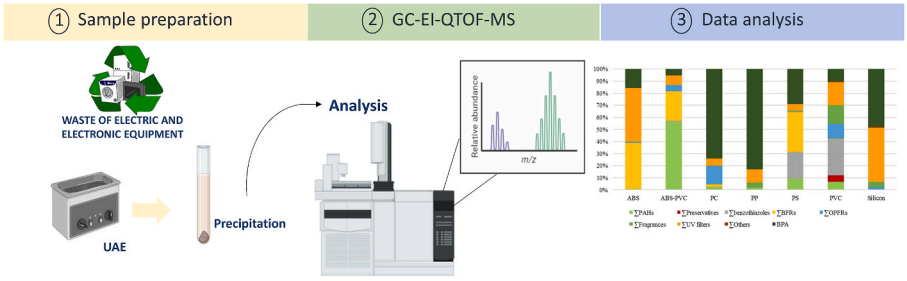
HIGHLIGHTS

- Non-target screening analysis for the identification of hazardous substances in WEEE.
- More than 300 volatile and semi-volatile substances were identified in WEEE plastic.
- Flame retardants, UV filters and PAHs exhibited the highest detection frequencies.
- TBBPA and TPhP displayed the highest concentrations.

GRAPHICAL ABSTRACT



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ABSTRACT

End-of-life electric and electronic devices stand as one of the fastest growing wastes in the world and, therefore, a rapidly escalating global concern. A relevant fraction of these wastes corresponds to polymeric materials containing a plethora of chemical additives. Some of those additives fall within the category of hazardous organic compounds (HOCs). Despite the significant advances in the capabilities of analytical methods, the comprehensive characterization of WEEE plastic remains as a challenge. This research strives to identify the primary additives within WEEE polymers by implementing a non-target and suspect screening approach. Gas chromatography coupled to time-of-flight mass spectrometry (GC-QTOF-MS), using electron ionization (EI), was applied for the detection and identification of more than 300 substances in this matrix. A preliminary comparison was carried out with nominal resolution EI-MS spectra contained in the NIST17 library. BPA, flame retardants, UV-filters, PAHs, and preservatives were among the compounds detected. Fifty-one out of 300 compounds were confirmed by comparison with authentic standards. The study establishes a comprehensive database containing *m/z* ratios and accurate mass spectra of characteristic compounds, encompassing HOCs. Semi-quantification of the predominant additives was conducted across 48 WEEE samples collected from handling and dismantling facilities in Galicia. ABS plastic demonstrated the highest median concentrations, ranging from 0.154 to 4456 μg g^{-1}, being brominated flame retardants and UV filters, the families presenting the highest concentrations. Internet router devices revealed the highest concentrations, containing a myriad of HOCs, such as tetrabromobisphenol A (TBBPA), tribromophenol (TBrP), triphenylphosphate (TPhP), tinuvin P and bisphenol A (BPA), most of which are restricted in Europe.

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1. Introduction

Electrical and electronic equipment (EEE) have become essential in our daily lives. The production of EEE remains as one of the fastest growing global manufacturing activities (Buekens and Yang, 2014), mainly fuelled by rapid technological advances and accelerated obsolescence. This translates into a rapid growth of waste of electrical and electronic equipment (WEEE), also known as e-waste. WEEE is defined as a complex mixture of plastic, metals and metalloid materials (Runde et al., 2022), which contains both valuable and hazardous substances that require special handling and recycling to avoid environmental contamination and detrimental effects on human health (Robinson, 2009). Mobile phones, televisions, laptops, or any equipment with a battery or an electrical power supply is considered as EEE, so they are likely to swell the mountain of WEEE at the end of their useful life. In 2019, 53.6 million tons of WEEE were generated worldwide (Global E-waste Statistics Partnership, 2020), of which only 17.4% was collected and recycled responsibly, while the rest was sent and, illegally dumped in low- or middle-socioeconomic countries (Global E-waste Statistics Partnership, 2020). To date, recycling to recover reusable components as Cu and precious metals from WEEE is the ultimate goal (European--Parliament, 2020). However, due to the limited number of facilities and high costs, landfilling, incineration, and exportation to poor countries remain as the main alternatives. These practices carry an associated environmental and human risk due to the presence of toxic substances (Das et al., 2021; Zeng et al., 2021). Thus, current European Directive on WEEE is under revision to ensure the protection of the environment by preventing, or reducing, the adverse impacts of the generation and the management of WEEE and a new Directive is planned to be released this year 2024. In the meantime, the scientific community efforts are focused on deepening the knowledge of plastic chemicals to bring together relevant information to finally establish an informed policy in the context of plastics-related processes. Thus, a new report on this topic has recently been published (Wagner et al., 2024).

Present European Union (EU) Directives require an important reduction of the WEEE plastic (WEEEP) landfilled, as this material is estimated to constitute 20.6 % of total European WEEE (Buekens and Yang, 2014). Nevertheless, one of the main limitations to achieve an actual sustainable circular economy is the lack of information regarding the chemical additives used in the different plastics and applications (Wagner et al., 2024). Among the different plastic polymers, the most commonly used in WEEE are acrylonitrile butadiene styrene (ABS), polystyrene (PS) (or high impact PS- HIPS), polycarbonate (PC), polypropylene (PP), polyethylene (PE), polyvinylchloride (PVC) and combinations of them (Buekens and Yang, 2014; Jia et al., 2022). As for the additives, many substances are added as pigments, polymer fillers, plasticizers, antistatic agents, flame retardants, stabilizers as thermic or UV filters, and reinforcing glass or carbon fibres (Buekens and Yang, 2014). Thus, containing many hazardous organic compounds (HOCs), many of them included in the restriction of organic hazardous substances (ROHs) directive, such as polybrominated biphenyls (PBB) and polybrominated diphenyl ethers (PBDEs) (Bill et al., 2022; European Parliament, 2011). In particular, great attention has been paid to halogenated flame retardants (HFRs), such as tetrabromobisphenol A (TBBPA) and hexabromocyclodecane (HBCD) (Kajiwara et al., 2011; Kousaiti et al., 2020; Sindiku et al., 2015; Yu et al., 2017). Information on the occurrence of other families of potentially HOCs is still limited, which is a key for improving reutilization.

Analytical approaches for the determination of metals in WEEEP are mainly based on the use of spectrometric techniques such as inductively coupled plasma optical emission spectroscopy (ICP-OES), ICP coupled to mass spectrometry (ICP-MS) and atomic absorption spectroscopy (AAS) (Andrade et al., 2022; Ángel Aguirre et al., 2013; Dimitrakakis et al., 2009; Duarte et al., 2010; Guimarães Araújo et al., 2012; Shi et al., 2021; Zeng et al., 2021), however methodologies focused on the comprehensive identification of organic additives remain limited. WEEEP is a complex matrix, generally composed of different polymers, so sample preparation and determination techniques oriented to non-target screening analysis are challenging. Due to this variety, certified reference materials are scarce, which further complicates the validation of the proposed methodologies. Common analytical strategies applied to complex solid matrix, such as liquid-solid extraction (LSE), Soxhlet, and ultrasonic-assisted extraction (UAE) are usually employed as extraction techniques for plastic materials, using solvents that swell and/or dissolve both the target analytes and the plastic, followed by the precipitation of the polymeric matrix (Ballesteros-Gómez et al., 2014; Cobo-Golpe et al., 2024; Silva et al., 2006). At this step of the analytical process, the selection of an appropriate solvent is the key step to successful results, due to transformation or degradation risks of some compounds during extraction, non-quantitative recoveries and interferences with other species generated from partial degradation of the parent polymers skeleton.

High resolution/accurate mass spectrometry (HRMS), either based on time of flight (TOF) or Orbitrap mass analysers are essential for non-target screening analysis, as they provide spectral information of any substance present in the sample amenable to chromatographic separation and ionization steps. When it comes to the analysis of volatile and semi-volatile compounds, such as phenols or halogenated flame retardants, gas chromatography -electron ionization (GC-EI) coupled to HRMS constitutes the preferred technique, as it provides reproducible and robust fragmentations (Hollender et al., 2017). Generally, accurate *m/z* values for fragment and molecular ion (when observed), are specific enough for the unambiguous identification of a given compound and no MS/MS data is required. Identification of *unknowns* is based on a database containing spectra for a high number of substances. NIST contains low (nominal) resolution spectra for more than 15,000 compounds (NIST v17). To date, the main disadvantage of this technique is the lack of accurate EI-MS spectral libraries available (Castro et al., 2019; Hollender et al., 2017), however as EI-MS spectra are more reproducible than MS/MS and libraries contains a higher number of spectra, spectral comparison with EI-MS, especially when including retention index information, constitute an excellent approach to reach tentative identifications (Hollender et al., 2017).

Herein, the aims of the present study are to 1) investigate the possibilities of a non-target screening approach, based on accurate electron ionization mass spectrometry (EI-MS) spectra, for the identification of volatile and semi-volatile compounds present in WEEEP collected from different handling facilities; 2) to create a database containing the *m/z* and spectra of the common chemical additives present in WEEEP and 3) to evaluate the concentrations of the identified substances, with particular attention to those considered as hazardous species based on their environmental toxicity, persistence and/or mobility. To the best of the authors' knowledge this is the first study aiming a non-target screening of organic additives in WEEEP samples and presenting an in-depth analysis of their occurrence.

2. Materials and methods

2.1. Chemicals and materials

LC-MS grade methanol (MeOH, 99.8%), dichloromethane (DCM, 99.8%) and iso-octane (i-Octane, 99%) were purchased from Merck (Darmstadt, Germany). Ammonium fluoride (NH_4F) was acquired from Sigma Aldrich (Milwaukee, WI, USA). Ultrapure water (18.2 mΩ cm^{-1}) was obtained from a Milli-Q system by Millipore (Billerica, MA, USA).

Standards of tentatively identified analytes in WEEEP samples, were acquired from Sigma-Aldrich and TCI Europe (Zwijndrecht, Belgium). Isotopically labelled standards (either deuterated or ^{13}C species) were obtained from Sigma Aldrich and Toronto Research Chemicals (North York, ON, Canada), Table S1. Individual stock solutions of each compound were prepared in MeOH and further dilutions were made in the same solvent. These compounds were first employed to assess the

performance of the analytical methodology and later as surrogate standards (SSs) to improve the reliability of the quantitative data provided for a selection of additives in WEEEP.

2.2. Waste sampling

Electronic and electrical waste samples were collected from 3 different waste handling facilities located in Galicia (Northwest of Spain) during winter 2023. The facilities included specialized sorting based on the different WEEE categories according the WEEE Directive 2012/19/EU (Council of the European Union, 2012) (Table 1). Samples were collected manually by representatively subsampling large piles of specific waste type using gloves. The selection of WEEE samples included small appliances containing different types of plastics. Once in the laboratory, samples were disassembled to recover plastic components (casings, fans, keyboards, etc). These components were rinsed with ultrapure water, allowed to dry at room temperature and finally ground below 2 mm using a mill (Retsch® SM100, Biometa) to reach their homogeneity. Those samples that were not possible to grind in the mill due to melting or rigidity, were broken down using a conic drill. The homogenized materials were stored in glass jars in a refrigerator at 4 °C until analysis. More information about the samples is presented in Table 1.

2.3. Sample preparation

A representative sample of WEEEP (≈0.3 g of ground plastic) was weight out and placed into a 15 mL polypropylene tube (PP). The sample was fortified with 40 μL of a mixture of SSs (50 μg mL^{-1}). Samples were extracted through ultrasound assisted extraction (UAE) with 8 mL of DCM (30 min, 35 °C). After extraction, the polymeric matrix was precipitated with 2 mL of MeOH (Yao et al., 2023) and centrifuged for 10 min at 4000 rpm. The obtained supernatant was collected and transferred to a clean PP tube (≈8 mL). Thereafter, 1 mL of extract was filtrated (hydrophobic filter 0.45 μm) and concentrated below 0.2 mL under a gentle nitrogen stream (N_2). Finally, samples were reconstituted in 1 mL of i-octane and ready for GC-MS analysis, or in 1 mL of MeOH, in case of TBBPA quantification by UPLC-MS.

2.4. Instrumental analysis

2.4.1. GC-QTOF-MS

WEEEP extracts were analysed using a gas chromatography hybrid quadrupole time-of-flight (GC-QTOF) MS instrument, purchased from Agilent (Wilmington, DE, USA), which consist of a 7890A gas chromatograph and a 7200 QTOF MS spectrometer, furnished with an EI source. The system was operated in 2 GHz mode (mass resolution (FWHM) of 6500 at *m/z* 131 Da). In case of non-target screening (NTS) and suspected target approaches, the MS spectra were recorded in profile mode (required for spectral deconvolution) at 2.5 Hz (5430 transients per spectrum), in the range of *m/z* values from 50 to 700 Da, while in target experiments, MS spectra were recorded in centroid mode. The *m/z* axis was automatically re-calibrated by infusion of perfluorotributyl amine (PFTBA) every 6 injections. Chromatographic separation was carried out in a HP-5 MS capillary column (30 m × 250 μm i.d., 0.25 μm film thickness) acquired also from Agilent. Helium was employed as carrier gas at a constant flow of 1.2 mL min^{-1}. The temperature of the oven was programmed as: 90 °C (1 min), rated at 8 °C min^{-1} to 300 °C (15 min). Standards and sample extracts (2 μL) were injected in the pulsed splitless mode (25 psi, 1 min), with the injector temperature set at 300 °C. The split flow and the splitless time were 60 mL min^{-1} and 1 min, respectively. The transfer line and the EI source temperatures were 280 °C and 230 °C, respectively. The temperature of the quadrupole (Q) MS analyser was 150 °C and the system was employed in the MS mode, with the Q serving as ion guide to the TOF.

Table 1
Overview of the samples analysed in this study.

ID	Type of plastic	WEEE category[b]	Description
AC-001	ABS[a]	IT and telecommunication equipment	Headset
AC-002	ABS[a]	IT and telecommunication equipment	Headset
AC-003	PS	IT and telecommunication equipment	Computer speaker
AC-004	ABS-PVC[a]	IT and telecommunication equipment	Headset
BAS-001	PS	Small household appliances	Scale
CAL-001	ABS[a]	IT and telecommunication equipment	Desk calculator
CB-001	PVC rigid + phthtalate[a]	Cable plastic	Cable plastic
CB-002	PVC rigid + phthtalate[a]	Cable plastic	Cable plastic
CB-003	PVC rigid + phthtalate[a]	Cable plastic	Cable plastic
CB-004	PVC rigid + phthtalate[a]	Cable plastic	Cable plastic
COND-001	Silicon[a]	IT and telecommunication equipment	Conductivimeter
DEP-001	ABS	Small household appliances	Hair remover
DIS-002	ABS[a]	IT and telecommunication equipment	Floppy disk
DIS-004	PS[a]	IT and telecommunication equipment	Floppy disk
DNI-001	ABS[a]	IT and telecommunication equipment	ID reader
IMP-001	ABS	IT and telecommunication equipment	Printer
IMP-003	PS-HI	IT and telecommunication equipment	Printer
IMP-005	ABS-FR	IT and telecommunication equipment	Printer
INT-001	PC-ABS	IT and telecommunication equipment	Ethernet connection station
LU-001	PC[a]	Lighting equipment	Emergency lights
LU-002	PC[a]	Lighting equipment	Emergency lights
MAN-001	ABS	IT and telecommunication equipment	Controller
MAN-002	Silicon[a]	IT and telecommunication equipment	Controller
PC-001	ABS	IT and telecommunication equipment	Computer
PC-002	PC[a]	IT and telecommunication equipment	Laptop
PL-001	PP[a]	Small household appliances	Iron machine
PL-002	ABS-nylon[a]	Small household appliances	Hair straightener
RAD-001	ABS	Large household appliances	Electric radiator
RAD-002	PP[a]	Large household appliances	Electric radiator
RAT-001	PC-ABS[a]	IT and telecommunication equipment	Computer mouse
RAT-004	ABS	IT and telecommunication equipment	Computer mouse
RD-001	SB	IT and telecommunication equipment	Radio
RD-002	ABS-PVC[a]	IT and telecommunication equipment	Radio
RD-003	ABS[a]	IT and telecommunication equipment	Radio
ROU-001	ABS y PC-ABS	IT and telecommunication equipment	Router
ROU-002	ABS y PC-ABS	IT and telecommunication equipment	Router
ROU-003	ABS[a]	IT and telecommunication equipment	Router
ROU-004	ABS	IT and telecommunication equipment	Router

(continued on next page)

Table 1 (*continued*)

ID	Type of plastic	WEEE category[b]	Description
SEC-001	PC-nylon[a]	IT and telecommunication equipment	Hair dryer
SEC_002	ABS-PC[a]	Small household appliances	Hair dryer
TEL-001	ABS	IT and telecommunication equipment	Phone
TEL-002	ABS	IT and telecommunication equipment	Phone
TEL-003	ABS[a]	IT and telecommunication equipment	Phone
TEL-004	ABS	IT and telecommunication equipment	Phone
TEL-005	ABS-PVC[a]	IT and telecommunication equipment	Phone
TEL-006	ABS	IT and telecommunication equipment	Phone
TV-001	PS[a]	IT and telecommunication equipment	TV
VENT-001	PC[a]	IT and telecommunication equipment	Computer fan

ABS (acrylonitrile butadiene styrene); PS (polystyrene); PVC (polyvinyl chloride); PC (polycarbonate); PP (polypropylene).

[a] Plastic analysed through ATR analysis.

[b] Categories according the WEEE Directive 2012/19/EU.

2.4.2. UHPLC-QqQ-MS

Quantification of TBBPA was performed by ultraperformance liquid chromatography tandem mass spectrometry (UPLC-MS/MS), using an Acquity UPLC system connected to a Xevo TQD triple quadrupole mass spectrometer, furnished with a Z spray ESI source, both acquired from Waters (Milford, MA, USA). Chromatographic separation was carried out in a Zorbax Eclipse Plus C_{18} Rapid Resolution HD column (2.1 × 50 mm, 1.8 μm) connected to a C_{18} 2.1 mm i.d. Security Guard™ ultracartridge, supplied by Agilent and Phenomenex (Torrance, CA, USA), respectively. Mobile phases were ultrapure water (A) and MeOH (B), both containing 1 mM NH_4F, at a constant flow rate of 0.4 mL min^{-1}. The gradient of mobile phase was programmed as follows: 0–1.30 min, 2% B; 1.31–2.50 min, 50% B; 2.51–6.00 min, 100% B; and 6.10–7.00 min, 2% B. Column and pre-column were maintained at 40 °C. The injected volume for solvent standards and sample extracts was 0.5 μL. TBBPA was ionized under ESI negative mode. Two transitions were monitored per compound (multiple reaction monitoring mode, MRM) considering a time window of 60 s around the retention time (RT), being the quantification transition (Q1) 542.8 → 447.8 (CE 34V) and qualification transition (Q2) 542.8 → 419.8 (38 V) for TBBPA; and Q1 552.8 → 454.8 (35 V) and Q2 552.8 → 80.9 (50 V) for TBBPA-d_{10}. The capillary and cone voltage were optimized as 3.50 kV and 27 V, respectively.

2.5. Qualitative and quantitative data analysis

The performance of the employed methodology was first validated in terms of extraction and determination for a selection of 18 isotopically labelled standards, belonging to different chemical families recognized as common additives in plastic from WEEE. A pooled WEEEP sample was prepared by mixing 7 different samples containing different plastic types. The recoveries (R%) of the sample preparation for the labelled compounds were estimated by dividing the response (peak area for each analyte) obtained in the pooled sample spiked before the treatment (pre-extraction) by the corresponding response of the same analyte obtained in a sample extract fortified at the same level after the sample treatment (post-extraction) and multiplied by 100. Potential matrix effects (MEs, %) during GC-MS analysis were assessed and estimated as the ratio between the analyte response obtained in a sample extract fortified after the treatment (post-extraction) and the response of the same compound for a solvent (i-octane)-based standard of the same concentration and multiplied by 100. Therefore, values below 100 % indicate signal suppression, while values above 100% indicate enhancement of the instrumental sensitivity during analysis. Ratios with values in proximity to 100% suggest the absence of ME (Raposo and Barceló, 2021). The instrumental limits of quantification (iLOQ) were calculated for each analyte as the concentration of the lowest calibration standard concentration providing a signal-to-noise (S/N) ratio of 10 or, alternatively, 10 times the standard deviation of the instrumental blanks divided by the slope of the calibration curve if the analyte was detected in them.

The NTS approach was applied to a set of 20 samples. Data mining was performed following a workflow previously described by the authors (Castro et al., 2019) with minor modifications (Fig. 1). Briefly, raw GC-EI-QTOF-MS files, acquired in profile mode, were loaded into Unknowns Analysis software and peak picking was performed with the *SureMass* algorithm, limiting the filtration to entities with an absolute peak height above 2000 counts. The deconvolution of the chromatograms was based on four different retention time windows (25, 50, 100 and 200 %) around the average peak width in each record. Spectra of deconvoluted features were compared to those compiled in the NIST17 library to determine the similarity to known spectra. The comparison algorithm was a combination of forward-reverse search (Blum et al., 2019), values of 0 and 1 corresponding to pure reverse and forward modes, respectively. Herein, a weight factor of 0.7 was set. A PCDL library, including linear retention index (LRI) and experimental accurate EI-MS spectra of all the injected standard solutions was created, independently of whether compounds were finally confirmed in the samples, or not.

As a result, a list of tentatively identified compounds was created and a set of 51 potentially hazardous compounds was selected for quantification. Quantification was performed using matrix-matched standards, containing the same concentrations of isotopically labelled SSs as those added to WEEEP samples before the extraction (Castro et al., 2019).

2.6. Quality assurance and quality control (QA/QC)

QA/QC proceedings were implemented during sample preparation and analysis to avoid potential contamination problems and to reduce the number of false positives and false negatives. Thus, all PP material was discarded after use. In addition, one procedural blank (without sample) was analysed per batch of 10 samples. Any signal in the blanks were subtracted from the samples as it could be assumed to be contamination valid for all the samples. SSs were spiked to the samples to account for any signal losses during sample preparation and instrumental analysis, and ten samples were analysed (including extraction) in duplicate for analytical method performance purposes. To monitor instrumental sensitivity deviations and carry-over effects during analysis, solvent blanks and standards (50 ng mL^{-1}) were analysed every 10 samples and a mass calibration was performed after 6 injections.

2.7. Software and spectral libraries

GC-QTOF-MS data was acquired with Agilent enhanced Mass Hunter GC-MS acquisition software, while peak inspection of raw chromatograms and quantification was performed with *Mass Hunter Qualitative* software (vB.08.00) and *Mass Hunter Quantitative* (v10.2), respectively. Spectral deconvolution was achieved with *Unknown Analysis* software (based on the *SureMass* algorithm), integrated in the *Mass Hunter Quantitative* software (version B.08.00). The creation of a custom-made spectral library (PCDL) was managed using also dedicated functions in the *Mass Hunter* software. Preliminary tentative identifications of the deconvoluted components in the GC-QTOF-MS files, were obtained using the NIST17 EI-MS library. The *MS Search* (v. 2.3) software was employed to manage spectra compiled in this library, and to calculate the theoretical *m/z* ratios of fragment ions with known structures in the NIST17 library. UPLC-MS/MS data were acquired with the MassLynx v4.1 software, and quantification of TBBPA in plastic extracts was performed with TargetLynx (Waters, Milford, U.S.). Excel (Microsoft 365)

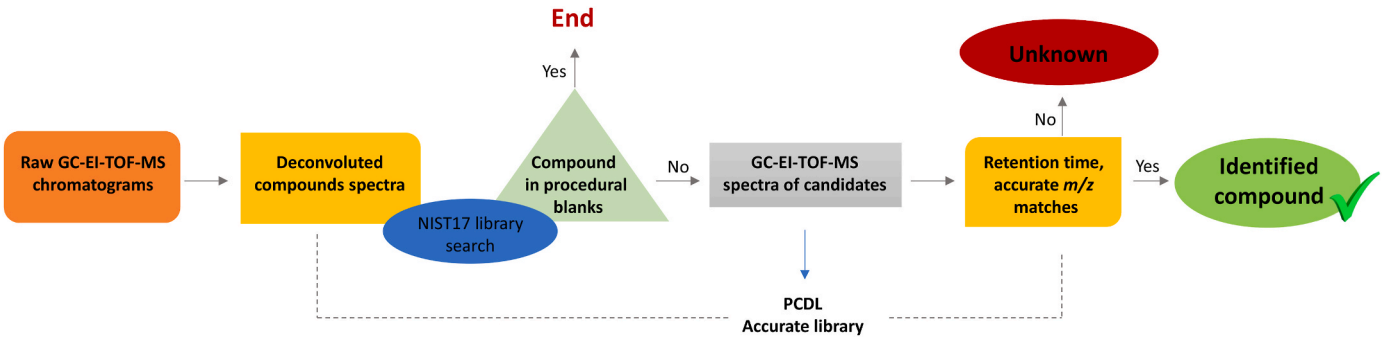

Fig. 1. Workflow followed during the non-target screening of HOCs in WEEEP (Figure adapted from (Castro et al., 2019)).

was used to perform data analysis.

3. Results and discussion

3.1. WEEEP characterization

Samples were categorized according to the Directive 2012/19/UE (Council of the European Union, 2012) as i) large household appliances; ii) small household appliances; iii) IT and telecommunication equipment; iv) consumer equipment and photovoltaic panels; v) lighting equipment; vi) electrical and electronic tools; vii) toys, leisure and sports equipment; viii) medical devices; ix) monitoring and control instruments; and x) automatic dispensers (Table 1). The type of plastic material was determined by attenuated total reflectance (ATR) with infrared (IR) spectroscopy and Raman IR (laser wavelength 532 and 785 nm), when it was not specified in the plastic cover of the product. Information on the instrument and main features of the methodology are summarized in Text S1. Reference ATR-IR and RAMAN-IR spectra were found in the IRUG spectral database available online (IRUG). The most characteristic absorption bands at the different wavelengths were detected in the experimental spectra and compared with those compiled in the database in order to elucidate the type of polymers present. Examples of experimental and database IR spectra leading to the identification of the polymers ABS and silicon are presented in Fig. S1 (A and B). Thus, the presence of ABS, PVC, HIPS, PC, silicon, and combinations of different types of plastic (copolymers), were verified in the selected samples (Table 1), being ABS the main component in most of the WEEEP analysed. As for the case of copolymers, they were classified in the category of the predominant plastic. According to Jia et al. ABS accounts for 30 % of the WEEP applications, followed by HIPS, which accounts for 25 %, while the rest are usually PC (10 %), ABS/PC blends (9 %) and PP (8 %) (Jia et al., 2022). Among the main characteristics of ABS, this polymer possesses great toughness and chemical resistance by a low prize, which might explain their widespread use (Olivera et al., 2016). This variety and combination of polymers difficult sample preparation, since each polymer presents a different behaviour in contact with the organic solvents tested during extraction of organic additives and HOCs. Table S2 summarizes the stability of different polymers vortexed for 1 min and ultrasonicated during 30 min, at 35 °C, as function of the extraction solvent. In those conditions, ABS and PS were completely dissolved in acetone, ethyl acetate, and DCM. On the other hand, PP, PVC, and silicon were more resistant to the organic solvents, being not affected under the abovementioned conditions. From these data, DCM and MeOH were selected for extraction and polymer re-precipitation, respectively. In this way, bonded and non-bonded additives existing in the polymers were transfer to the organic extract.

3.2. Characterization of the sample preparation methodology

Sample preparation methodology was evaluated in terms of extraction efficiency and matrix effects for a set of 18 isotopically labelled compounds belonging to different chemical families (Table S1). R % for isotopically labelled compounds were evaluated at one concentration (200 ng g^{-1}) in triplicate, and ranged between 67 % and 84 %, with RSD< 41 %, while values of ME % indicates enhancement of the signal for most of the considered chemicals (Table S1). As previously described by Meng et al. (2020), during plastic analyses many components are often co-eluting, leading to poor quantitation accuracy. However, herein signal enhancement is likely related to the injection processes, rather than to variations in the efficiency of compounds ionization at the EI source. Anyway, the large variability in the extraction efficiency observed for some of the isotopically labelled model compounds might be also related to non-quantitative extractions and losses during polymer re-precipitation and solvent-exchange. These MEs were especially problematic in ABS and PVC plastic, thus quantitation with matrix-matched calibration is highly recommended (Raposo and Barceló, 2021).

Instrumental repeatability (n = 3) and reproducibility (n = 3 days) were assessed by analysing replicates of 200 ng mL^{-1} standard solvent solution; and the obtained results demonstrated RSDs <14 and 36 %, respectively.

3.3. Non-target screening analysis of WEEEP samples

A set of 20 samples of WEEEP products were extracted and analysed via GC-QTOF-MS (section 2.5.). A total of 305 candidates presented high spectra similarity, displaying normalized scores (0–100) above 60, which can be considered either level 2 (probable structure) or level 3 (tentatively identified) (Schymanski et al., 2014). Among these candidates, those presenting mass errors below 5 mDa, detection frequencies (DF%) above 5% and that were not detected, or detected with higher intensities than in procedural blanks, were tentatively identified. Compounds commonly known for blank contamination are bisphenol A (BPA), phthalates and the organophosphate flame retardant (OPFRs), triphenylphosphate (TPhP) (Brandsma et al., 2022; Castro et al., 2019). In the present study, blank contamination was carefully investigated. BPA, TPhP and tinuvin 329 were detected in the blanks, although presenting significantly lower signals than in the organic extracts from plastic items.

Commercial standards of 51 out of 305 candidates, together with other compounds belonging to the same families or plastic additives commonly reported in literature due to their occurrence, persistence and toxicity (i.e., organophosphate flame retardants, phthalates, …) were analysed in the GC-EI-TOF-MS for spectral and retention time comparison with those corresponding to the obtained deconvoluted spectra (maximum allowable differences equal to 5 mDa and 0.1 min, respectively), and incorporated in a customized library, also containing their CAS numbers, retention times and LRI values.

3.4. Suspect screening and semi-quantification of organic additives and HOCs by GC-EI-TOF-MS

A customized EI-MS library containing 339 compounds with their corresponding accurate spectra, which included 51 of the tentatively identified substances, was applied for the suspect screening of 48 WEEEP samples. The accurate EI-MS library is available in Zenodo repository (https://zenodo.org/; https://doi.org/10.5281/zenodo.10045296). The abovementioned strategy applied during the NTS was also followed in the suspect screening analysis. Thus, deconvolution and library search of the obtained spectra were carried out. Compound identification was achieved by matching all the peaks in the experimental spectra with the spectra in the customized EI-MS library, limiting the identification results to a minimum match factor of 60 %, accurate mass tolerance below 50 ppm and a chromatographic retention time window of 40 s. The identification of tribromophenol (TBrP) in ABS plastic using the accurate EI-MS library is illustrated in Fig. 2. Extracted ion chromatogram and mass spectrum of TBrP (M^+ 329.7682 and retention time 12.436 min) are presented in Fig. 2A and B, respectively. Mass spectrum was compared to the spectra contained in the EI-MS library (Fig. 2C). Thus, differences between calculated and experimental *m/z* for the most intense fragment ions were calculated and stayed below 2.3 mDa (Table 2). The accurate experimental spectrum for a spiked pool of WEEEP confirmed the identity of the candidate as TBrP. Following this workflow, a total of 79 compounds were tentatively identified in the studied samples (Table 2).

Table 2 compiles a list of compounds detected in WEEEP following the abovementioned methodology, including their use, detection frequency (DF %), calculated mass errors (mDa) and confirmation with commercial standards. Most of the candidates can be categorized as flame retardants (FR), PAHs, personal care products (PCPs), pesticides, plasticizers, and UV filters (Table 2). Information on the chemical composition of WEEEP is still scarce, only a few authors have previously performed suspect screening of plastic consumer products and plastic from WEEE toys (Lowe et al., 2021; Meng et al., 2020; Wagner et al., 2024), reaching the same conclusions as those presented herein: PAHs, pesticides, bisphenols and FR (OPFRs and BFRs) are the families displaying the highest detection frequencies (DF %) in this type of matrix (Table 2). Other authors have addressed the occurrence of FRs, such as polychlorinated biphenyl substances (PCBs) (Yu et al., 2017), new brominated flame retardants (NBFRs) and organophosphorus flame retardants (OPFRs) (Ballesteros-Gómez et al., 2014; Bill et al., 2022; Roth et al., 2012), and other contaminants of emerging concern (CECs) such as bisphenols, benzophenone UV filters (Runde et al., 2022), and per- and polyfluoroalkyl substances (PFAS) (Tansel, 2022). The presence of NBFRs (Lan et al., 2023), PFAS (Zhang et al., 2020), phthalates (Li et al., 2023), bisphenols (Wei et al., 2023), fluorinated biphenyls and analogues (Zhu et al., 2021), benzothiazoles and benzotriazoles (Li et al., 2020) was also reported in dust and soils from WEEE dismantling areas, pointing out that this waste might constitute an emission source of the abovementioned chemicals.

As for the semi-quantification, a total of 51 analytes were considered (Table S3). Most of the studied compounds presented corrected R % between 60 % and 100 %, with RSD <28 %. In the case of ME %, following the same tendency as their deuterated analogues, enhancement of the signal was observed for most of the considered analytes (Table S3). The linear response range for every analyte was assessed by the injection of solvent-based standards at seven concentrations ranging from 20 ng mL^{-1} to 500 ng mL^{-1}. Within this interval, the obtained calibration curves fitted a linear model, presenting determination coefficients (R^2)> 0.99 for the majority of the analytes. The iLOQs were also calculated displaying values between 0.70 and 567 ng mL^{-1} (Table S3).

3.5. Determination of TBBPA by UPLC-QqQ-MS

The quantification of TBBPA constitutes a challenge due to its medium polarity (logK_{ow} 5.9) and presence of two phenolic moieties in the molecule (Liu et al., 2016; Sunday et al., 2022). GC-MS analysis requires a previous derivatization step, i.e. using methyl chloroformate, to improve the detectability and the reproducibility of the analysis (Covaci et al., 2009; Sunday et al., 2022). The obvious result is an increase in the complexity of the analytical procedure. Herein, target analysis of TBBPA was accomplished by UPLC-QqQ-MS, due to this technique provides higher sensitivity and lower iLOQs. The suitability of the proposed methodology was demonstrated through the evaluation of R % and ME % (Table S3), providing values of 74 % (RSD 19 %) and 80 % (RSD 13 %), respectively. Obtained iLOQ of TBBPA was 2.04 ng mL^{-1}.

3.6. Occurrence of organic additives and HOCs in WEEEP

Forty-eight out of 51 targeted compounds were detected in the different samples. Most of them are compounds of environmental concern due to their persistence, toxicity, mobility (i.e. benzotriazole)

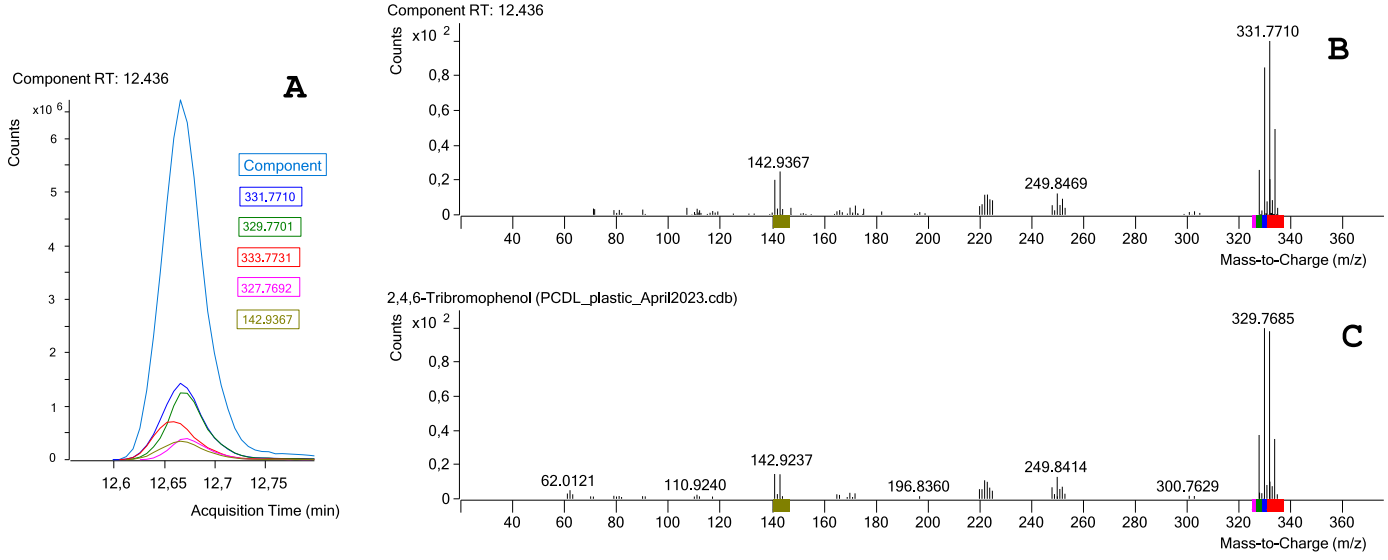

Fig. 2. Extracted ion chromatogram (EIC) of TBrP (A) for a non-spiked sample (RAT-001). Spectra of the deconvoluted component (B) and the experimental accurate spectra of TBrP (C).

Table 2
Tentatively identified compounds in the extracts from plastic samples by GC-QTOF-MS.

Compound								Other ions		
	Use/Type	Retention time (min)	CAS number	Database *m/z*	Experimental *m/z*	Mass error (mDa)	DF (%)	Ion 2	Ion 3	Confirmed with standard
BHT	Antioxidant	19.48	128-37-0	205.16	205.1595	−0.50	35%	177.1285		✓
3,5-Di-*tert*-butyl-4-hydroxybenzoic acid (BHT-COOH)	Antioxidant	23.62	1421-49-4	235.1682	235.1717	3.50	5%	250.1918		✓
2,6-Di-*tert*-butyl-4-hydroxy-4-methyl-2,5-cyclohexadien-1-one (BHT-OH)	Antioxidant	25.25	10396-80-2	219.1443	219.1385	−5.80	20%	191.1132		✓
Antioxidant 2246	Antioxidant	37.17	119-47-1	340.2404	340.2408	0.40	10%	177.1282		
Pentachloroanisole	Bactericide	24.25	1825-21-4	264.8338	264.8369	3.10	15%	236.8373		✓
Triclosan	Bactericide	31.91	3380-34-5	287.9494	287.9525	3.10	5%	218.0123	145.9685	✓
Nicotine	Drug	15.37	54-11-5	161.1073	161.1038	−3.50	5%	84.0808	119.0612	✓
Octanoic acid	Fatty acid	10.64	124-07-2	73.0292	73.027	−2.20	30%	101.0605	60.0215	
Nonanoic acid	Fatty acid	13.15	112-05-0	73.0293	73.027	−2.30	15%	115.0762		
dodecanoic acid	Fatty acid	20.56	143-07-7	73.0291	73.0271	−2.00	5%	129.092		
TCEP	Flame retardant	25.13	115-96-8	248.9853	248.9877	2.40	30%	204.9587	142.9661	✓
TCPP	Flame retardant	25.93	13674-84-5	125.0001	125.0006	0.50	10%	201.0083	277.0165	✓
Triphenyl phosphate (TPhP)	Flame retardant	36.85	115-86-6	325.0723	325.0638	−8.50	50%	215.0275	169.0658	✓
Cresyl diphenylphosphate (CDP)	Flame retardant	38.20	26444-49-5	340.0871	340.0876	0.50	5%	165.0709		✓
2,3′,4′,6-Tetrabromodiphenyl ether (BDE 71)	Flame retardant	38.71	189084-62-6	485.7114	485.7111	−0.30	5%	325.877		✓
EHDPP	Flame retardant	38.91	1241-94-7	251.0481	251.0482	0.10	15%			✓
BDE-99	Flame retardant	42.59	60348-60-9	403.7863	403.7877	1.40	5%	563.6223		✓
Tetrabromobishenol A (TBBPA)	Flame retardant	46.05	79-94-7	528.7309	528.7286	−2.30	10%	447.8116	272.8718	✓
Methyl anthranilate	Flavouring Agent	15.21	134-20-3	119.0377	119.0383	0.60	5%	151.0642		
Vainillin	Flavouring agent	16.65	121-33-5	151.0395	151.0383	−1.20	35%	123.0443		✓
1,1′-Biphenyl	Flavouring agent or adjuvant	16.17	92-52-4	154.0772	154.0758	−1.40	25%	76.0309		
Benzaldehyde, 4-methoxy-	Flavouring Agents	12.94	123-11-5	135.0444	135.0407	−3.70	10%	107.0497		
a-Methylstyrene	Flavouring agent	6.05	98-83-9	118.0772	118.0754	−1.80	30%	103.0542		
alpha-amylcinnamaldehyde-isomer2	Flavouring agent	22.60	122-40-7	129.0694	129.07	0.60	5%	115.0539	202.1347	
alpha-hexylcinnamaldehyde	Flavouring agent	24.78	101-86-0	129.0699	129.0678	−2.10	10%	115.0542	216.1503	
alpha-hexylcinamaldehydeisomer2	Flavouring agent	24.78	101-86-0	129.0695	129.0705	1.00	15%	115.0544	216.1501	
Diphenyl ether	Flavouring agent and fragrance	16.75	101-84-8	170.0725	170.0721	−0.40	5%	141.0701		
Cinnamal	Flavouring agent and Pesticide	13.36	104-55-2	131.0491	131.0488	−0.30	15%	103.0539		
Longifolene	Fragrance	16.94	475-20-7	161.1343	161.1324	−1.90	40%	91.0551		
Hedione	Fragrance	22.73	24851-98-7	83.0501	83.0479	−2.20	10%	153.1286		
α-Guaiene	Natural product	16.84	3691-12-1	105.0718	105.0682	−3.60	15%	91.0559		
Caffeine	Natural product	26.82	58-08-2	194.0809	194.0805	−0.40	5%			✓
N-Nitrosodiphenylamine	Nitrosamine	21.99	86-30-6	169.0885	169.0854	−3.10	5%	83.5364		
Naphthalene	PAH	11.09	91-20-3	128.0622	128.0593	−2.90	20%	99.0461		✓
Acenaphthene	PAH	18.79	83-32-9	153.0701	153.0684	−1.70	5%			✓

(*continued on next page*)

Table 2 (*continued*)

Compound	Use/Type	Retention time (min)	CAS number	Database *m/z*	Experimental *m/z*	Mass error (mDa)	DF (%)	Other ions		
								Ion 2	Ion 3	Confirmed with standard
Fluorene	PAH	21.11	86-73-7	166.0776	166.0775	−0.10	5%	139.0542		✓
2,6-Diisopropylnapthalene	PAH	24.28	24157-81-1	197.1316	197.1317	0.10	40%	212.1558	155.0853	✓
Phenanthrene	PAH	25.44	85-01-8	178.0781	178.0777	−0.40	15%	152.0619		✓
Pyrene	PAH	31.05	129-00-0	202.0781	202.0802	2.10	5%			✓
Coumarin	PCP	17.67	91-64-5	146.0362	146.036	−0.20	5%	118.0412		
Methyl paraben	PCP	18.04	99-76-3	121.0299	121.0272	−2.70	5%	152.0467		✓
Ethyl paraben	PCP	19.76	120-47-8	121.0301	121.0243	−5.80	20%	166.0623		✓
Propyl paraben	PCP	21.98	94-13-3	121.0294	121.0255	−3.90	10%	138.0312		✓
Ethyl-4-dimethylaminobenzoate	PCP	23.96	10287-53-3	193.1092	193.1078	−1.40	55%	148.0753	164.0702	
Galaxolide	PCP	27.01	1222-05-5	243.1747	243.1764	1.70	5%	213.1633		✓
Tonalide	PCP	27.28	21145-77-7	243.1753	243.1772	1.90	5%	187.1127		✓
2,6-dimethylphenol	Pesticide	9.00	576-26-1	122.0724	122.0709	−1.50	15%	107.0489		
2,5-Dichlorotoluene	Pesticide	9.42	19398-61-9	159.9872	159.9832	−4.00	5%	125.018		
2,4-Dichlorophenol	Pesticide	10.60	120-83-2	161.9652	161.9625	−2.70	10%	97.9941		
2,3,5-Trichlorophenol	Pesticide	15.46	933-78-8	195.9298	195.9246	−5.20	15%	159.9526	96.9879	✓
2,4,6-Tribromophenol	Plasticizer	22.32	118-79-6	329.7685	329.7708	2.30	15%	140.9277		✓
Benzothiazole	Plasticizer	12.16	95-16-9	135.0145	135.013	−1.50	60%	108.0034		✓
Phthalic anhydride	Plasticizer	14.52	85-44-9	104.0227	104.0246	1.90	20%	76.0282		✓
Dimethyl phthalate	Plasticizer	18.06	131-11-3	163.0422	163.0388	−3.40	15%	133.0306		✓
Diethyl phthalate	Plasticizer	21.39	84-66-2	149.0234	149.0241	0.70	70%	121.0283		✓
2-Hydroxy-benzothiazole	Plasticizer	22.91	934-34-9	151.009	151.0092	0.20	20%	96.0027	123.0138	✓
N-butyl benzenesulfonamide	Plasticizer	25.52	3622-84-2	170.0276	170.0299	2.30	15%	141.001		
Dibutyl phthalate	Plasticizer	29.10	84-74-2	149.0234	149.0218	−1.60	25%	121.0309		✓
Bisphenol A	Plasticizer	33.49	80-05-7	213.0944	213.0908	−3.60	10%	119.0517		✓
Tributyl Acetylcitrate	Plasticizer	34.57	77-90-7	185.0769	185.0816	4.70	15%	129.015		
Benzyl butyl phthalate	Plasticizer	36.01	85-68-7	149.0232	149.0228	−0.40	5%	91.0537		✓
Bis(2-ehtylhexyl) adipate	Plasticizer	36.77	103-23-1	129.0545	129.052	−2.50	5%	111.0438	70.0767	
Dicyclohexyl phthalate	Plasticizer	39.09	84-61-7	149.0264	149.0226	−3.80	40%	167.0385		
Di-(2-ethylhexyl) terephthalate	Plasticizer	41.98	6422-86-2	261.1533	261.1533	0.00	35%	149.027	167.0378	✓
Di-isononyl phthalate	Plasticizer	42.76	20548-62-3	149.0284	149.0219	−6.50	10%	293.1807		✓
Diisodecyl phthalate	Plasticizer	44.55	89-16-7	149.0285	149.0248	−3.70	20%	307.1969		✓
Benzophenone	UV filter	22.18	119-61-9	182.0728	182.0706	−2.20	50%	105.0338	77.0385	✓
Homosalate	UV filter	27.66	118-56-9	120.0218	120.0186	−3.20	5%	138.0324		✓
Benzophenone 3	UV filter	30.43	131-57-7	227.0696	227.0713	1.70	5%	151.0383		✓
Tinuvin P (drometrizole)	UV filter	31.09	2440-22-4	225.0913	225.0865	−4.80	5%	168.0827		✓
EHMC	UV filter	35.50	5466-77-3	178.0639	178.0659	2.00	15%	161.0609		✓
Tinuvin 350	UV filter	38.51	36437-37-3	308.1797	308.1762	−3.50	5%	238.0996		✓
Tinuvin 326	UV filter	39.17	729335	300.0925	300.0921	−0.40	5%	272.0609		✓
Tinuvin 329	UV filter	39.52	52188-76-8	252.113	252.1144	1.40	5%	133.0608		✓
Tinuvin 328	UV filter	40.75	25973-55-1	322.1946	322.1924	−2.20	20%	252.1152		✓
Tinuvin 327	UV filter	40.88	3864-99-1	342.138	342.1364	−1.60	20%	286.0759		✓
Octocrylene	UV filter	41.00	6197-30-4	248.0713	248.0731	1.80	35%	204.0837	360.1972	✓
4,7-Methanoazulene, 1,2,3,4,5,6,7,8-octahydro-1,4,9,9-tetramethyl-, [1S-(1. alpha.,4.alpha.,7.alpha.)]-		16.05	514-51-2	105.0716	105.0684	−3.20	25%	161.1353		

and ubiquitous character. Identifications and concentrations in duplicate and reinjected samples were self-cross checked, presenting RSD <36 % (n = 10 duplicates). Total mean concentrations in the different types of plastics ranged from 13.9 μg g^{-1} to 7218 μg g^{-1}, being PP and ABS, the polymers containing the lowest and the highest concentrations for this set of hazardous semi-volatile compounds, respectively (Table 3 and Fig. 3A). Among the different families, the plasticizer bisphenol A (BPA) presented the highest concentrations in PC and PP, accounting for the 74 % and 83 % of the total concentration, respectively (Fig. 3B). UV filters and BFRs were predominant in ABS and silicon plastic (Fig. 3B).

3.6.1. BPA

BPA is one of the most abundant synthetic chemicals in the world (Usman and Ahmad, 2016). This substance is commonly used as monomer during the synthesis of PC plastics and epoxy resins. Runde et al. previously reported the concentrations of BPA in WEEE samples collected in Norway, reporting median concentrations of 22 μg g^{-1} (Runde et al., 2022) and similar median concentrations (71 μg g^{-1}) were also previously reported by Arp et al., in 2017 (Arp et al., 2017) for WEEE samples. In the present study, median concentrations of 34.5 μg g^{-1} were measured for BPA. The complete set of sample concentrations is presented in Table S4.

3.6.2. HFRs

BFRs, both PBDEs and NBFRs, were detected in the samples, being TBrP and TBBPA those showing the higher prevalence, with DF of 21 % and 38%, respectively (Table S4). TBrP is commonly used as pesticide and flame retardant in thermoplastics and epoxy resins, ABS and PS (Norwegian Environmental Agency, 2016). Herein, TBrP was mostly detected in ABS plastic and copolymers in median concentrations of 11.6 μg g^{-1}. As for TBBPA, 70–90 % of the produced substance is used as a reactive flame retardant in epoxy, PC and phenolic resins in printed circuit boards, while the rest is commonly used as additive in ABS plastic and phenolic resins (Fjäder et al., 2022; Liu et al., 2016). In the latter ones, TBBPA is not chemically bound to the material, so it can be easily released to the environment. Many authors reported concentrations in WEEE and dust from dismantling areas. Yu et al. studied the occurrence of BFRs in housing plastics from different types of WEEE, reporting concentrations from n.d. to 34.0 μg g^{-1} (Yu et al., 2017). Lower concentrations were presented by Kousaiti et al., indicating that TBBPA concentrations vary significantly within the same plastic type, whose values stayed between < LOQ and 3.11 μg g^{-1} (Kousaiti et al., 2020). Herein, TBBPA was detected in ABS and PC plastic, presenting higher concentrations than reported in literature and ranging from 23.8 ng g^{-1} to 20.7 mg g^{-1} (median 269 ng g^{-1}). Routers from different production years and brands were found to contain the highest TBBPA concentrations, ranging from 0.16 μg g^{-1} for the newest acquired item (year 2021) to 20.7 mg g^{-1} for the oldest (year 2002), while PBDEs remained non detected. This trend was previously reported by Bill et al., who compared PBDE and TBBPA median concentrations in EEE samples

Table 3
Summary of the main descriptive statistics (detection frequency (DF, %), mean, median, min and max) of the concentrations (ng g^{-1}) obtained for the different HOCs families in WEEE samples (n = 48 samples).

Type of plastic	Descriptive statistics	∑PAHs	∑Preservatives	∑benzothiazoles	∑BFRs	BPA	∑OPFRs	∑Fragrances	∑UV filters	∑Others	∑TOTAL
ABS (n = 24)	***DF (%)***	88%	25%	4%	58%	75%	83%	4%	83%	4%	96%
	mean	1106	597	808	3861249	1588439	72084	308	4455753	154	7218092
	median	420	811	808	1194	48401	1238	308	13983	154	78765
	min	61	63	808	24	5455	140	308	228	154	0
	max	5143	923	808	20749247	27826282	1283173	308	88278244	154	117387859
ABS-PVC (n = 3)	***DF (%)***	100%	0%	33%	33%	100%	100%	67%	100%	0%	100%
	mean	642	–	1927	56369	12434	11048	525	17832	–	61737
	median	600	–	1927	56369	5938	5357	525	2054	–	13456
	min	535	0	1927	56369	5510	511	521	803	0	8373
	max	790	0	1927	56369	25853	27276	529	50639	0	163383
PC (n = 7)	***DF (%)***	86%	29%	14%	43%	86%	71%	29%	100%	0%	100%
	mean	7594	630	823	15159	559865	116331	1043	47117	–	623697
	median	4008	630	823	7319	147990	74882	1043	28074	–	179212
	min	542	269	823	68	69260	2460	879	3778	0	114592
	max	18021	990	823	38089	2496584	306930	1206	123744	0	2826710
PP (n = 2)	***DF (%)***	100%	0%	0%	0%	100%	0%	50%	100%	0%	100%
	mean	269	–	–	–	11877	–	608	1547	–	13997
	median	269	–	–	–	11877	–	608	1547	–	13997
	min	222	0	0	0	8346	0	608	179	0	12185
	max	317	0	0	0	15409	0	608	2915	0	15810
PS (n = 5)	***DF (%)***	100%	0%	20%	60%	40%	60%	20%	100%	0%	100%
	mean	1846	–	9031	13446	11916	176	308	2171	–	18825
	median	1884	–	9031	75	11916	115	308	1668	–	4139
	min	590	0	9031	27	9237	94	308	616	0	2475
	max	3523	0	9031	40235	14595	318	308	4211	0	70290
PVC (n = 4)	DF (%)	100%	50%	75%	25%	25%	100%	100%	100%	100%	100%
	mean	10915	9672	53555	319	18581	21834	27321	34333	615	144744
	median	7389	9672	390	319	18581	23345	11894	25943	733	104893
	min	2363	189	281	319	18581	13857	1355	5470	45	67439
	max	26518	19155	159995	319	18581	26787	84141	79975	949	301750
SB (n = 1)	DF (%)	100%	100%	0%	0%	0%	100%	0%	100%	0%	100%
	mean	1366	41651	–	–	–	23	–	628	–	43668
	median	1366	41651	–	–	–	23	–	628	–	43668
	min	1366	41651	0	0	0	23	0	628	0	43668
	max	1366	41651	0	0	0	23	0	628	0	43668
Silicon (n = 2)	DF (%)	100%	0%	0%	0%	50%	100%	100%	100%	100%	100%
	mean	291	–	–	–	34549	1309	3229	32047	624	54776
	median	291	–	–	–	34549	1309	3229	32047	624	54776
	min	43	0	0	0	34549	382	805	7268	341	8838
	max	540	0	0	0	34549	2237	5654	56827	907	100714

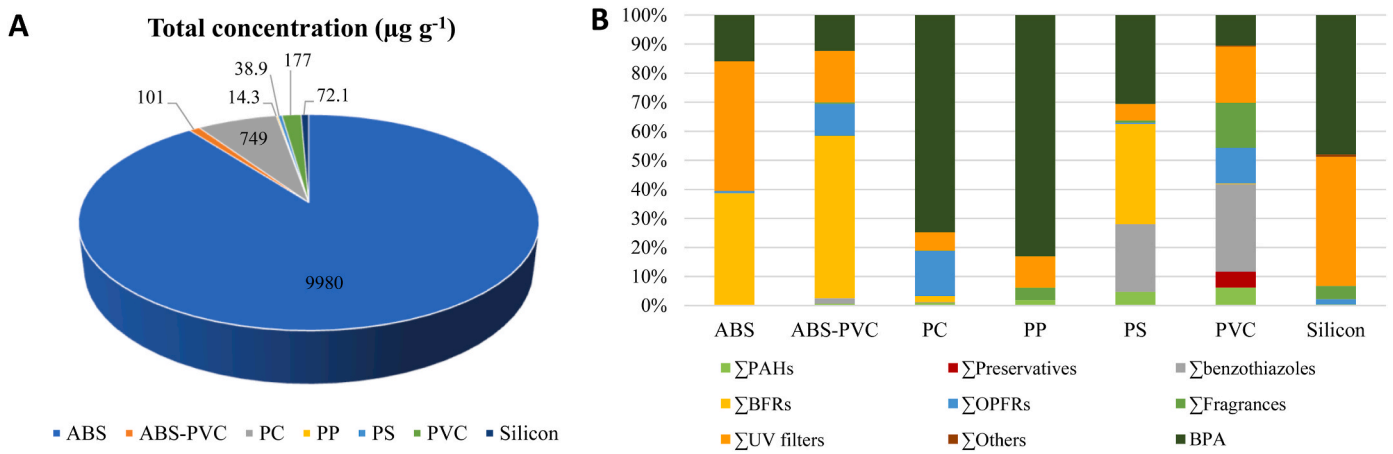

Fig. 3. Total mean concentration of organic additives and HOCs in different type of plastic (A) and percentage of the different families in each plastic (B).

manufactured between 2009 and 2017. The authors found negative correlations between PBDEs and TBBPA concentrations, with PBDE concentrations being higher in older products, while TBBPA concentrations increased in newer products (Bill et al., 2022). This may be explained by the restriction of PBDEs, indicating that this phase-out is being compensated by the application of TBBPA. To the best of the authors knowledge, this extremely high TBBPA concentrations in router devices is reported for the first time in literature. Occurrence of TBBPA was evaluated also in water, soil and biological samples from a WEEE dismantling area in China (Wei et al., 2023), demonstrating the widespread occurrence of TBBPA in the studied samples, presenting concentrations up to 33.8 ng g^{-1} in soil samples and DF of 100% (Wei et al., 2023). Considering the very high concentrations of TBBPA and its potential migration from the plastic into the surrounding environment, monitoring of these substances in WEEEP is therefore essential.

Organophosphate flame retardants (OPFRs) are chemicals widely used in consumer products to delay combustion processes or prevent fire (Castro et al., 2023; Li et al., 2019). Halogenated OPFRs, such as TClPP or TDClPP are mostly used as flame retardants, while the non-halogenated ones are common plasticizers (van der Veen and de Boer, 2012). In the present study, OPFR concentration was controlled by two species, TClPP and TPhP. TClPP was mainly detected in cable plastic in concentrations between 1.83 and 25.89 µg g^{-1}, while TPhP was ubiquitous in the studied samples (DF 73 %) from <iLOQ to 1215 µg g^{-1} (median concentration of 1.05 µg g^{-1}). Occurrence of OPFRs was evaluated in consumer products in 2011 by Kajiwara et al. (2011). According to their results, TPhP presented the highest concentrations in all the studied samples, ranging from 560 ng g^{-1} to 14,000 µg g^{-1}. TBBPA and TClPP were the HFRs presenting the highest concentrations, but in line with the results both additives are used in different applications. According to Fig. 4, it seems clear that both compounds are not used combined.

3.6.3. UV filters

UV filters are organic chemicals used in plastic materials to prevent the degradation caused by UV light (Yao et al., 2023). Herein, UV filters were detected in 92 % of the analysed samples, presenting mean concentrations between 179 ng g^{-1} and 88,278 µg g^{-1}, being Tinuvin P and Tinuvin 326 detected in the highest concentrations, 1908 µg g^{-1} (mean concentration and 56 % DF) and 1784 µg g^{-1} (42 %), respectively (Fig. 5). Fig. 5 summarizes UV filter concentrations, presenting average, maximum and median concentration. Literature on UV filters in WEEE is still scarce, to the authors knowledge, only Runde et al. assessed the occurrence of benzophenone UV filters in WEEE and vehicle fluff (Runde et al., 2022). The authors detected median concentrations between 2.00 and 104 ng g^{-1}. Herein, median concentrations of benzophenone 3 (BP3) were higher than reported by Runde et al. (≈40 ng g^{-1}). These lower concentrations can be derived from the different type of plastic

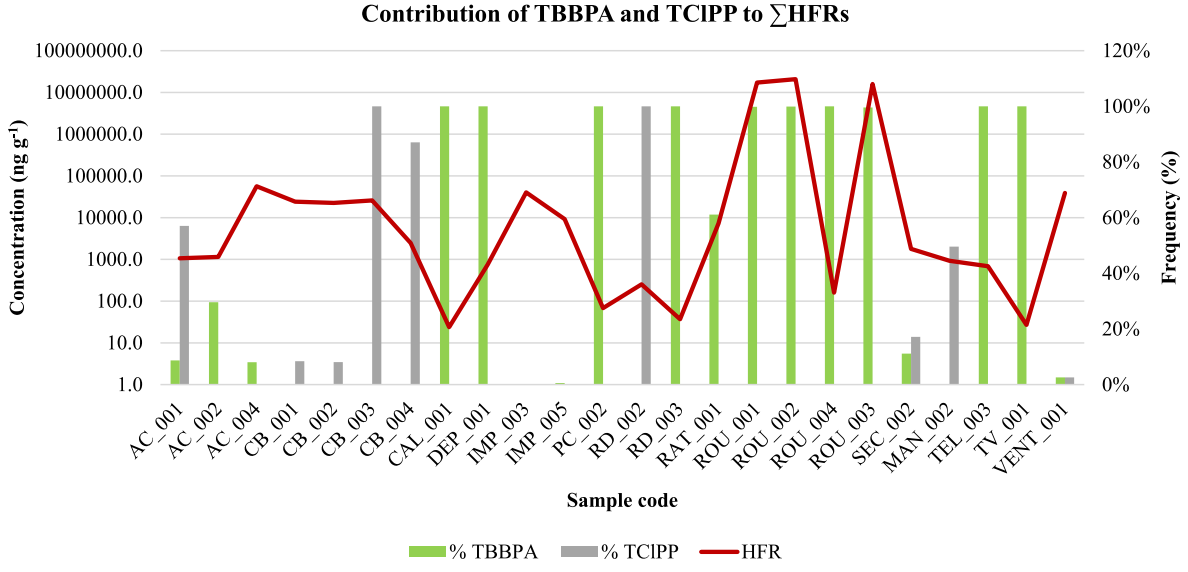

Fig. 4. Contribution of TBBPA and TClPP to the total concentration of HFRs.

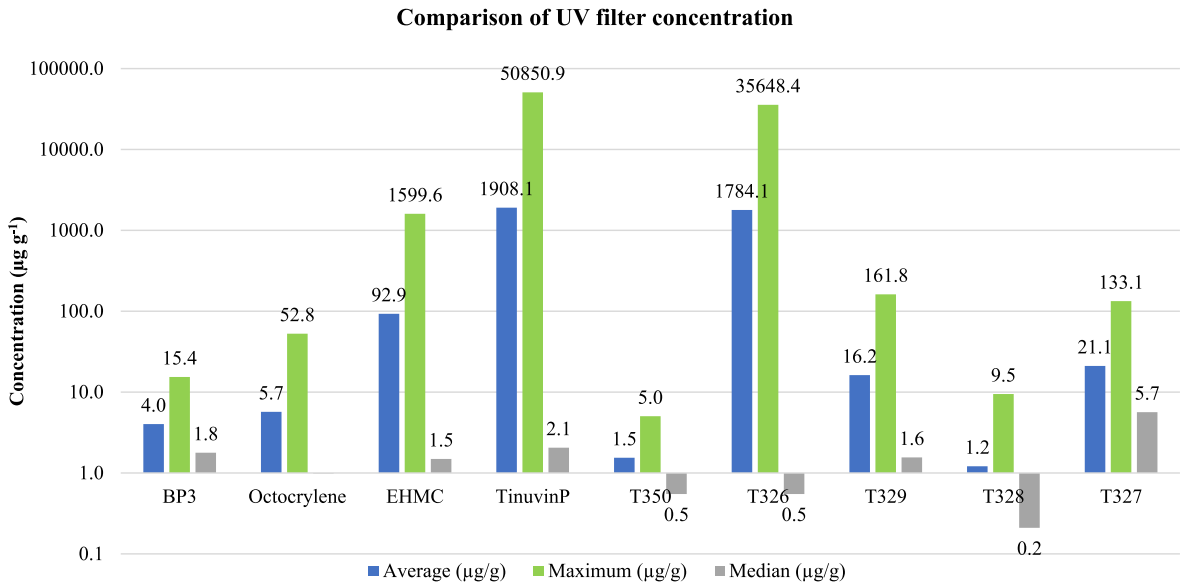

Fig. 5. Summary of concentrations (µg g^{-1}) for selected UV filters in WEEEP samples.

analysed. Concentrations of UV filters were recently evaluated in biodegradable plastic products by Yao et al. (2023), demonstrating the same tendency than reported here, the highest concentrations detected for Tinuvin P and Tinuvin 326.

3.6.4. PAHs and preservatives

PAHs were mostly detected in ABS plastic. $\sum_{11}$PAHs ranged from 0.043 µg g^{-1} to 26.51 µg g^{-1} (median concentration of 0.83 µg g^{-1}) (Table S4), being phenanthrene and anthracene the substances presenting the highest mean concentrations, 920.7 ng g^{-1} (38 %) and 627.2 ng g^{-1} (42 %), respectively. To the best of the author's knowledge, PAHs in WEEEP are reported for the first time, so comparison with previous studies is not possible. However, many authors described the occurrence and fate of these substances in the environment derived from electronic waste dismantling activities (Chen et al., 2019; Ma et al., 2022), reporting total PAHs concentrations between 25 ng g^{-1} to 37,000 ng g^{-1} (d.w.), and demonstrating that significantly higher levels of PAHs were observed in particulate matter compared with sites no affected by WEEE recycling facilities (Ma et al., 2022). PAHs are recognized as a by-product released during heat treatment (combustion or pyrolysis) of the WEEE plastic (Das et al., 2021).

As for preservatives, this family of compounds presented the lowest DF (2–6 %) apart from methyl paraben that presented DF of 21 % and median concentrations of 360 ng g^{-1}.

3.7. Environmental implications and impacts of HOCs in WEEE

The presence of organic additives and HOCs in WEEEP poses significant environmental implications and impacts that necessitate careful consideration. According to Andrade et al. more than 1000 organic and inorganic compounds are present in WEEE (Andrade et al., 2022), constituting a direct threat to environment and human health. This threat is mainly derived from i) the waste treatment and recycling methods, such as landfilling and incineration (Baldé et al., 2017), which are insufficient, and ii) the toxicity, persistence, and bioaccumulation of the organic additives and HOCs present in WEEEP. As for the treatment, due to these residues are commonly disposed into landfills or improperly managed, these substances are susceptible to leach into soil and water, accumulate in living organisms and potentially entering the food chain. In case of recycling, incineration of WEEE with energy recovery is the main alternative. However this process is considered ecologically unacceptable for most of plastic wastes, due to it can release toxic contaminants as dioxins to the atmosphere as a consequence of the thermal degradation of PAHs (Choi and Lee, 2007; Dempsey and Timethy Oppelt, 1993; Jia et al., 2022). Thus, the presence of organic additives and HOCs in WEEE hinders the recycling and recovery of valuable materials from electronic components and plastics. This results in the depletion of resources and exacerbates the overall environmental impact of the waste. Jia et al. elaborated on the reutilization of plastic from WEEE and reported that recycling can save up to 80 % energy compared to the production of virgin material. In addition, it can reduce CO_2 emissions by 2.5 Mt annually for every 9.5 Mt of WEEE recycled (Jia et al., 2022). However, to date, only recycling of HDPE and LDPE has been developed, while no solutions have been found for the main polymers used in WEEE (ABS and HIPS). Recycled WEEEP can be either used to produce new products (i.e. filler materials, floor carpets, pots, park benches, …) or reutilise in concretes, roads, or plastic lumbers, among other products in which the requirements of physical and chemical properties are not demanding (Jia et al., 2022; Kazemi Najafi, 2013; Parra et al., 2019; Shi et al., 2022). However, often manufacturing costs can be higher than the cost of producing new virgin materials. Any case, pretreatment of these polymers to remove some of these hazardous substances is highly advisable to limit the reintroduction of these chemicals in the environment (Xia et al., 2021). Therefore, addressing the implications and impacts of organic additives and HOCs in WEEE requires a multidisciplinary approach involving waste management practices, regulatory measures, and the development of safer alternatives to HOCs in electronic products. By doing so, the adverse consequences of these hazardous compounds on ecosystems, human health, and the environment can be minimized while promoting a more sustainable and responsible approach to WEEE disposal and recycling.

4. Conclusions

The analysis of plastics from WEEE remains largely unexplored, as the complexity and variety of polymers used makes sample preparation and analysis challenging. However, research into new methods of recycling these wastes to ensure a circular economy depends primarily on the knowledge of what substances are present.

Non-target screening analysis appears as a suitable approach. Herein, a non-target screening strategy was developed for the first time for the analysis of WEEEP samples by GC-QTOF-MS, being able to tentatively identify more than 300 substances. The substances identified belong to different families commonly used as plastic additives, flame

retardants, PAHs, plasticizers, and UV filters. A new database containing the *m/z* and the obtained spectra of 339 compounds was created and uploaded to Zenodo, being semi-quantified a total of 51 substances. BPA, flame retardants, UV filters, PAHs and preservatives were among the compounds detected, highlighting their prevalence in WEEEP samples. TBBPA and TPhP were the substances detected in the highest concentrations ranging from 14.0 μg g^{-1} to 7292 μg g^{-1}. For the first time, paramount residues of TBBPA are reported in the plastic cover of router devices, pointing out to the necessity of addressing the implications with comprehensive approaches, including improved waste management practices, regulatory measures, and the development of safer alternatives to HOCs in electronic products. By adopting these strategies, the adverse consequences of these hazardous compounds for ecosystems, human health and the environment might be mitigated.

CRediT authorship contribution statement

G. Castro: Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Resources, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **M. Cobo:** Writing – review & editing, Methodology, Investigation, Formal analysis. **I. Rodríguez:** Writing – review & editing, Validation, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemosphere.2024.141946.

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