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Preventing brominated flame retardants from occurring in recycled expanded polystyrene: comparing Norwegian visual sorting with advanced screening methods

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ABSTRACT

There is international interest to increase recycling rates of expanded polystyrene foam (EPS). Extensive use of brominated flame retardants (BFRs), however, presents a hinder to this. If uncontrolled, hazardous BFRs could persist in recycled EPS leading to new exposure routes, including in materials such as EPS packaging where no flame retardants are required. This study looked at EPS foam collected from Norwegian Municipal Waste Sorting Facilities, visually sorted as "white EPS foam", mostly derived from packaging. Bromine was analysed by X-ray fluorescence (XRF), and selected BFRs including hexabromocyclododecane (HBCDD) were analysed by targeted gas chromatography-mass spectrometry analysis. Results were compared with EU and UNEP low persistent organic pollutant concentration limits (LPCLs). One out of 120 samples contained HBCDD over established LPCLs, likely attributable to missorted insulation EPS. Further, no false negatives occurred, as all samples in which target BFRs were quantified had XRF-detectable bromine. Visual sorting of white EPS packaging foam, with the use of XRF in uncertain cases has the potential of minimizing hazardous BFRs in recycled EPS. The context of national sorting infrastructure and compliance should be a central feature of future studies investigating how BFRs or other hazardous substances enter the global circular economy.

1. Introduction

Expanded polystyrene (EPS) foam, having a global production of 10 million metric tons in 2018 (Garside, 2019), is widely used in the construction and packaging sectors (Abdallah et al., 2018). As a packaging material, EPS foam is lightweight, rigid and able to withstand heavy loads relative to its material composition (2% polystyrene, 98% air) (Samper et al., 2010). From a circular economy perspective, EPS foam represents an important waste stream for waste reduction, reuse, redesign and recycling (Korhonen et al., 2018; Kral et al., 2013). More efficient recycling strategies could also help reduce exposure of hazardous EPS litter (Turner, 2020). Recycling of EPS is technically possible, as methods are in place to convert EPS to its monomer styrene (Stenmarck et al., 2017).

Many EPS products contain hazardous brominated flame retardants (BFRs) like hexabromocyclododecane (HBCDD) (Turner, 2020). Due to its persistence, toxicity and ecotoxicity, HBCDD was in 2008 listed on the Europe Union (EU)'s REACH (Registration, Evaluation, Authorisation and Restriction of Chemicals (EC 1907/2006) Candidate List of substances of very high concern (SVHC) as a persistent, bioaccumulative and toxic (PBT) substance (Article 57d), and in 2013 it was added to the Stockholm Convention on POPs (UN, 2013). Further, the EU and United Nations Environment Programme (UNEP) have introduced a low persistent organic pollutant (POP) concentration limit (LPCL) of 0.1% by weight (or 1000 mg/kg) for recycling of certain brominated compounds, including HBCDD, as well as a limit of 0.01% (100 mg/kg) as an unintentional contaminant, including recycled materials, above which products are not permitted for sale (EU, 2016). EPS can also contain other BFRs like tetrabromobisphenol A (TBBPA), as well as the POP substance decabromodiphenyl ether (BDE-209) (Rani et al., 2014). A newer BFR additive that is currently more common in EPS is PolyFR (CAS No. 1195978-93-8), which is a copolymer of polystyrene and brominated polybutadiene. PolyFR is not currently considered a hazardous substance (Koch et al., 2019).

With the increased motivation to recycle EPS, there is a concern that phased-out or restricted BFRs may persist in recycled products, including those they were not intended for, like EPS packaging foam. Packaging foam has been found in some countries to contain high levels of HBCDD (Abdallah et al., 2018; Rani et al., 2014), potentially attributable to recycling practices. The presence of BFRs in recycled EPS materials

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introduces new exposure routes to humans, such as by dermal contact to the EPS and products packed within (Abdallah et al., 2018), in addition to environmental litter (Turner, 2020).

In Norway, approximately 70000 tons EPS is imported or produced yearly, of which about 50% is used as fish boxes, while most of the rest is used in the building and construction industry as isolation or foundations. Smaller amounts are used as transport protection for electronic products and furniture (AvfallNorge, 2020). The collection of EPS from fish boxes and other packaging in 2019 was 5353 tons (GPN, 2020).

The EPS foam waste from municipal households in Norway is dominated by white packaging. For disposal of this waste, Norwegian citizens are instructed to place small pieces of EPS waste into their normal household waste (typically incinerated for energy) (Arp et al., 2017), while larger pieces should be delivered to a municipal waste sorting facility (MWSF). These MWSFs have a designated container for white EPS, and these containers give instructions to dispose coloured EPS for either incineration or, in the case of EPS insulation plates, as hazardous waste (due to their likelihood of having BFRs).

In this study the presence of BFRs was investigated in white EPS collected from Norwegian MWSFs. The purpose was to see what level of chemical analysis is needed to prevent HBCDD and other BFRs from entering a potential recycling stream for packaging EPS above the LPCLs of 0.01% and 0.1%. The possible options in this respect was 1) no analysis, only visual sorting; 2) simple bromine screening (e.g. with portable X-ray fluorescence, XRF); 3) advanced bromine screening (e.g. XRF of dissolved EPS, following Schlummer et al. (2015)); and 4) targeted BFR substance specific analysis (e.g. gas chromatography-mass spectrometry, GC-MS).

2. Material and methods

2.1. Sampling and Municipal Waste Sorting Facilities

EPS samples (n = 120) were collected between February and March 2019 at four MWSFs from diverse locations in Norway. Site information is provided in Table 1. A randomised, representative collection of EPS packaging from designated sorting containers was collected, and the samples were placed in transparent LDPE bags, transported to NGI and kept sealed at room temperature until analysis.

EPS sampling was done to reflect the visual diversity of samples in the container, based on shapes (e.g. fish boxes, packaging, irregular, plates), colour (shades of white/miscolouration) and degree of weathering (see below). The diversity of packaging is considered to reflect recently disposed waste, based on communication with the MWSF staff, as the containers are generally filled on the scale of days to weeks depending on the location.

2.2. Chemicals

Pure analytical grade acetone (VWR, Darmstadt, Germany) and technical grade bromine (certified reference material Br-, 1000 ± 30 µg/l, Spectrapure Standards AS (Oslo, Norway)) was used to make four calibration standards with bromine concentrations ranging from 0 to 1000 mg/kg (0, 10, 100 and 1000).

2.3. Sample preparation

Prior to analysis, all samples were weighed and photographed. Colour, degradation/weathering degree (0 = not degraded; 1 = some degradation; 3 = massive degradation, Fig. 1) and, if possible, source, label, production year and country of origin was noted.

2.4. XRF analysis for detection of bromine in foam samples

All EPS foams (n = 120) were cut into approximately 10 × 10 x 10 cm pieces and analysed for bromine content using a mounted, hand-held XRF (X-ray fluorescence spectroscopy) apparatus (Thermo Scientific Niton XL3t). Each solid foam sample was scanned with XRF at three locations (60 sec. per scan) on the exterior and also on the freshly cut interior.

2.5. GC-MS analysis

Samples with detectable (n = 16) and a random selection of undetectable (n = 20) bromine levels (based on XRF analysis) were sent to the accredited laboratory Eurofins Environment Testing AS for analysis of BFRs (HBCDD, Penta-BDE (commercial mixture), Octa-BDE (commercial mixture), Deca-BDE (BDE-209) and TBBPA; LOQ = 10 mg/kg), to verify whether bromine could be an indicator of the presence of prioritized BFRs. In brief, the samples were extracted with dichloromethane with an internal standard mix in an ultrasonic bath. The extracts were further analysed with GC-MS (Agilent, column: 15 m DB-5 (J&W), 0.25 mm i.d., 0.1 µm film) using helium as carrier gas with a temperature programmable cold injection system (temperature program: 70 °C, 2 min. up to 345 °C final temperature).

2.6. XRF analysis for detection of bromine in acetone extracts

Schlummer et al. (2015) published a screening method for rapid field identification of HBCDD containing EPS (and extruded polystyrene, XPS), and further differentiation from PolyFR containing EPS. The test principle is based on PolyFR not being extractable in acetone whereas HBCDD is, along with other acetone-soluble BFRs. XRF analysis of acetone extracts was performed for a selection of samples with high levels of bromine in the solid phase (n = 3), based on XRF analysis of EPS samples (section 2.4) and samples with measurable HBCDD levels (based on GC-MS analysis, n = 1). Following Schlummer et al. (2015), 2 g EPS foam was put in a 500 ml glass beaker and 5 g acetone was added and the glass beaker was manually swirled until complete visual dissolution. After 5 min, the supernatant of about 3 g of clear acetone extract was transferred with a glass pipette into polyethylene sample cuvettes, covered with mylar film, and placed in the XRF stand for analysis. Each acetone sample was scanned with XRF at three locations (60 sec. per scan).

Table 1
Municipal Waste Sorting Facilities (MWSF) sampled and details from the expanded polystyrene foam (EPS) sampling campaign

MWSF	Number of samples (n) and sample IDs	Location	EPS waste characteristics	EPS waste amounts
Mile	n = 20 M-1 to M-20	Drammen municipality. Centrally located in the eastern and most populated part of Norway	Mostly white EPS packaging from households (detached houses)	250-300 kg is usually received per month (3000-3600 kg per year)
Lyngås	n = 20 L-1 to L-20			8000 kg white EPS was received in 2018
Haraldrud	n = 40 H-1 to H-40	Oslo city. Norway's capital and most populous city. Located in the eastern part of Norway	Mostly white EPS packaging from households (apartment buildings)	Not provided
Finnmark Ressurs	n = 40 F-1 to F-40	Hammerfest municipality. Located in the northern- and eastern-most county in Norway.	Mostly EPS fish boxes and white EPS packaging from industry and households	12 × 10^6 kg fish boxes per year, and 1600 kg white EPS packaging

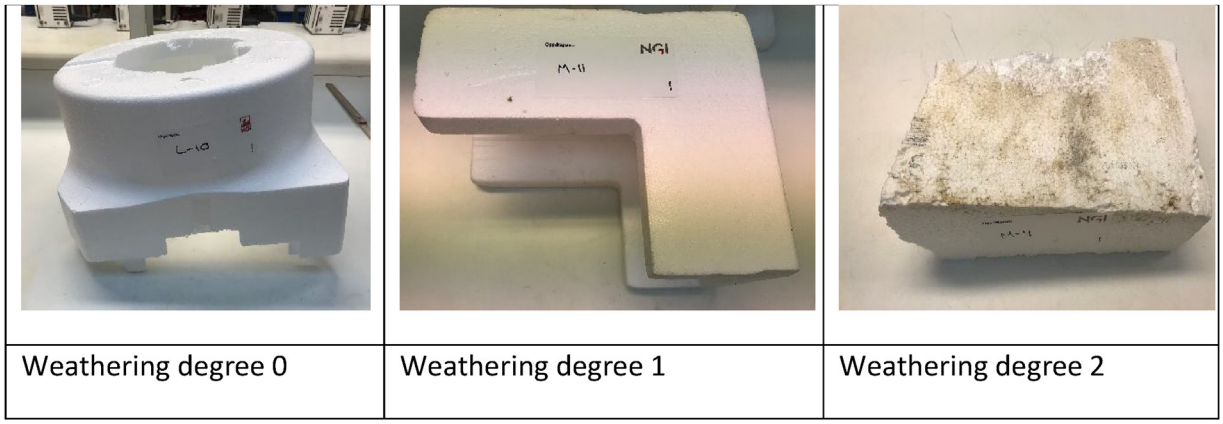

Fig. 1. Example of degradation/weathering degrees.

2.7. Quality control and quality assurance

The solid foam samples were scanned at the exposed surface as well as on the inner, sliced part, to verify if bromine was evenly distributed in the EPS (three scans per surface).

Quantification of bromine in the solid foam samples (LOQ of 8 mg bromine/kg) was carried out using the internal calibration of the instrument using three polyvinylchloride (PVC) standards (Thermo Scientific, Norway) with bromine levels of 0, 499 and 1099 mg/kg. The curve was highly linear (r^2 = 0.9996, Fig. S1).

Bromine levels in acetone extracts were calculated from the calibration curve obtained from the calibration standards of 0, 10, 100 and 1000 mg/kg (Section 2.2.). The curve was highly linear (r^2 = 0.9999, Fig. S2). The limit of quantification (LOQ, calculated as lowest calibration standard) was 10 mg/kg.

During the XRF measurements, a system check was carried out at the start and end of each day's measurement and at intervals of every hour.

The accredited laboratory Eurofins Environment Testing AS ensured GC-MS quantification over specific masses (m/z) were performed against external standard calibration and corrected using PCB 209 as an internal standard, with an estimated relative expanded measurement uncertainty of ± 14%.

3. Results and discussion

3.1. Bromine levels in solid EPS foam

Bromine was detected by XRF analysis in 16 out of 120 EPS samples (13%, Table 2), ranging from 9.4 to 4453 mg/kg (mean ± SD: 1500 ± 1900; median: 120, n = 16). Six of these had bromine levels >1000 mg/kg (range: 3413-4454). The remaining samples containing bromine (n = 10) ranged from 8.1 to 660 mg/kg (mean: 120 ± 190; median: 40). The difference between the two groups was significant (p = 0.00014, n = 120). There was no significant difference between bromine levels measured on the exposed surface vs. the interior (p = 0.96; Table S2), which indicate that bromine was evenly distributed throughout the samples. There were no significant difference in bromine content between samples of different weathering degree (p = 0.50-0.99, n = 120). However, most samples did not show any signs of weathering (Table S1), as most of the EPS packaging consisted of waste directly from households.

The highest bromine content was measured in sample M-13 (4500 mg/kg), which looked like an insulation plate, though was coloured white, unlike most such plates in Norway (often blue). EPS insulation materials often contain BFRs (Harrad et al., 2019; Sharkey et al., 2018). Relatively high bromine levels were also found in sample H-25 (3500 mg/

Table 2
Concentrations (mg/kg dry weight) of brominated flame retardants HBCDD and TBBPA measured with GC-MS, as well as bromine measured with XRF in EPS-samples from Mile (M), Lyngås (L), Haraldrud (H) and Finnmark (F). Penta-, octa and deca-BDEs were not quantifiable over the methods LOQ (10 mg/kg). LOQ for HBCDD and TBBPA (GC-MS): 10 mg/kg. LOQ for bromine in solid EPS samples (XRF) 8 mg/kg; LOQ for bromine in acetone extracts (XRF): 10 mg/kg. Only samples with measurable bromine and/or BFR levels are shown in the table ().

Sample-ID	HBCDD (GC-MS)	TBBPA (GC-MS)	Estimated bromine content based on target GC-MS[a]	Bromine (XRF)[b]	Acetone Extractable bromine (XRF)[c]
M-5	<LOQ	<LOQ	-	12	-
M-13	8900	<LOQ	6650	4500	700
L-2	<LOQ	<LOQ	-	9.4	-
L-7	<LOQ	<LOQ	-	640	-
H-4	<LOQ	14	8.2	3600	710
H-7	<LOQ	<LOQ	-	120	-
H-12	<LOQ	<LOQ	-	120	-
H-17	<LOQ	<LOQ	-	25	-
H-20	<LOQ	<LOQ	-	19	-
H-25	<LOQ	<LOQ	-	3600	-
H-35	<LOQ	<LOQ	-	86	-
H-36	<LOQ	<LOQ	-	4200	<LOQ
F-11	<LOQ	<LOQ	-	32	-
F-15	<LOQ	<LOQ	-	16	-
F-16	<LOQ	280	160	3500	-
F-38	<LOQ	200	120	3500	540

[a] Based on measured HBCDD or TBBPA concentrations multiplied by percent mass bromine of 0.747 and 0.588 respectively, for conversion to equivalent bromine concentration (estimate).
[b] XRF analysis on solid EPS foam; cXRF analysis of acetone extractable bromine.

kg), which was a white EPS sample with black dots. The black dots are likely due to manufacturing impurities of black EPS in white EPS (according to a personal communication with the Norwegian EPS producers society, *EPS-foreningen*). However, black dots do not necessarily imply bromine, as sample M-7 had a similar frequency of black dots, but with no measurable bromine with the applied XRF method. High bromine levels were also found in white EPS samples H-36 and H-4 (4000 and 3600 mg/kg, respectively), as well as F-16 and F38 (3500 mg/kg), which were not visually distinguishable from the others.

3.2. Comparison of XRF and BFR analysis

The BFR analysis by GC-MS of the 16 samples with bromine detected by XRF and 20 diverse, representative samples where bromine content was < LOQ are presented in Table 2 and Table S3. In none of the samples where bromine was < LOQ was a BFR quantified (Table S3), meaning there were no "false negatives" using XRF. HBCDD was detected above LOQ in only one of the 16 samples, and TBBPA above LOQ in three of the 16 samples. For these four samples, the GC-MS derived bromine content was calculated based on this data (Table 2). In general, XRF gave a higher bromine content than what was calculated based on targeted GC-MS results, and some of this discrepancy may be due to additional brominated compounds being present. In this study, there were eight samples containing bromine but none of the target POP-BFRs; however, these likely contain other flame retardants not measured by the GC-MS analysis in this study, including PolyFR as well as "emerging BFRs" (Nyholm et al., 2013; Rani et al., 2014), possibly also as a cocktail of non-target BFRs from PS/EPS recycling.

The sample with HBCDD detected was the suspected white insulation-plate M-13, with concentrations above the LPCL limit for hazardous waste (8900 mg HBCD/kg, Table 2). Harrad et al. (2019) presented that insulation plates in particular are dominated by HBCDD, and that hand-held XRF data generally correlate well with HBCDD for insulation plates as well as other construction and demolition waste. This also highlights the importance of separating such EPS waste (i.e. insulation plates) from packaging waste, for recycling. The three samples with TBBPA were H-4, F-16 and F-38. All of these were white EPS samples that resembled packaging material and could not be visually distinguished from other samples.

As shown in Table 2 and Fig. 2, acetone-extractable bromine was measured over the LOQ (10 mg/kg) in three of the four samples, with levels ranging from 540 to 710 mg/kg. The acetone extractable bromine was consistently less than the total bromine, indicating the extraction was not quantitative. For the sample with large amounts of HBCDD, M-13, only 15% of the bromine was transferred to the acetone fraction. For the three other EPS samples with high bromine content, bromine was transferred into the acetone fraction of two of the samples (H-4 and F-38), but not H-36 (Fig. 2). This latter case may be due to the use of brominated polymers or other products that are not acetone extractable, for example PolyFR (Schlummer et al., 2015). The former two cases are partly explained by trace levels of TBBPA from the GC-MS, but not quantitatively (Fig. 2). This implies the presence of one or several acetone-soluble BFRs that were not analysed for by GC-MS, potentially TBBPA derivatives like TBBPA-DPBE (Khaled et al., 2018), other BFRs (Zheng et al., 2019), or possibly breakdown products of PolyFR (Koch et al., 2016; Koch et al., 2019) and other BFRs.

3.3. Comparison with Literature

There were two other known studies that looked at BFR content in EPS packaging waste, a study from the UK and Ireland (Abdallah et al., 2018) and a South Korean study (Rani et al., 2014). In the UK and Ireland study, 27 out of 29 EPS packaging samples, mostly for food and electronic appliances, had HBCDD levels above a detection limit of >0.0007 mg/kg, with a mean value and range of 20 ± 25 mg/kg and 0.16-91 mg/kg, respectively. This can be considered low compared to the 100 mg/kg LPCL. The same study measured substantially higher concentrations of HBCDD in EPS packaging for lab equipment and construction materials (mean: 526 ± 1573 mg/kg, range: 0.036-5897 mg/kg, n = 14), which crossed the LPCL limit in some cases (Abdallah et al., 2018). In the South Korean study, two EPS packaging samples were analysed; an ice box for food packaging and electronic appliance packaging material. These had HBCDD levels of 960 and 0.65 mg/kg, respectively (Rani et al., 2014). Compared to this study's relatively high LOQ of HBCDD of 10 mg/kg, there were 15 out of 29 packaging samples that exceeded this in the UK and Ireland study, and one out of two in the South Korean study. By comparison, only one out of 120 (or 0.8%) had detectable HBCDD levels in this study – the suspected insulation plate. This could indicate that the "white EPS" in Norwegian sorting facilities may contain more virgin material, or recycled virgin material with less flame retardants.

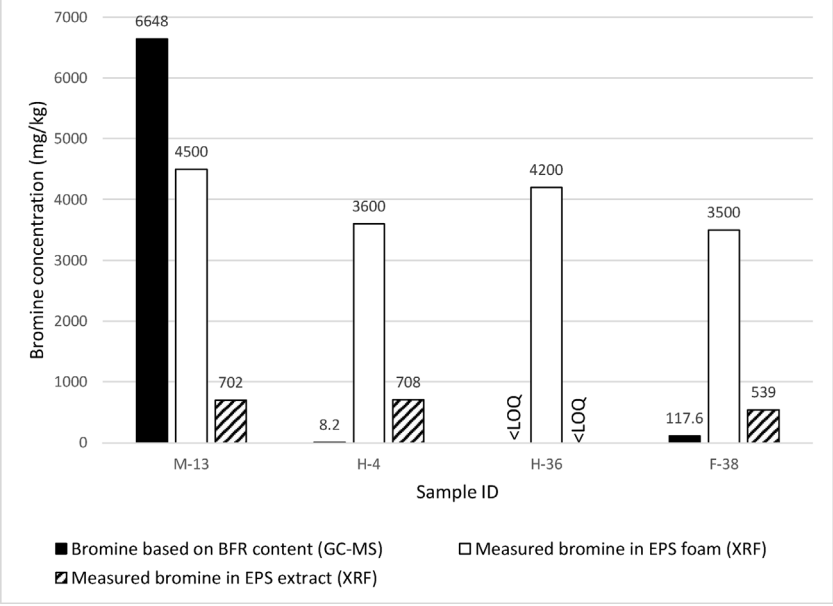

Fig. 2. Bromine content in samples M-13, H-14, H-36 and F-38 (based on bromine content measured by XRF in EPS foam samples and EPS foam extracts, and estimated bromine levels based on measured BFR content (GC-MS analysis)).

4. Recycling potential for Norwegian, white EPS packaging waste

Based on the XRF results of the solid EPS samples in this study (n = 120), the median concentration of bromine was 4 mg/kg, when half of the LOQ of 8 mg/kg was used in the calculations when the concentrations were < LOQ. Thus, as a conservative estimate, considering that in 2019, 5353 tons EPS waste packaging was collected for recycling in Norway (see above), the amount of bromine being sent into recycling would be 21 kg per year, or 0.0004 % the weight of recycled EPS. These results would favour recycling of sorted white EPS in Norway, as this is well below the LPCL value for BFRs of 0.1% (1000 mg/kg) for recycling and 0.01% (100 mg/kg) above which products are not permitted for sale. Only one sample out of 120 had HBCDD above the LPCLs, likely due to a missorted insulation plate sample. There were three samples having TBBPA, with only two exceeding 0.01%, and none had measurable levels of PBDE.

In this study we asked the question which level of options would be needed to be below the LPCL, with the options being 1) no analysis, only visual sorting; 2) simple XRF screening; 3) advanced screening using acetone dissolves subsamples; and 4) targeted BFR substance specific analysis.

It appears evident that in Norway the current visual sorting system used, to prevent coloured EPS, insulation EPS or small EPS pieces, to enter the white EPS packaging waste stream for recycling, appears an effective strategy. This study did not include packaging peanuts, EPS from laboratory ware or EPS from construction and demolition (except for one wrongly sorted insulation plate), but based on results from e.g. Abdallah et al. (2018), Drage et al. (2018) and Harrad et al. (2019), we recommend that these be not included for recycling, unless a recycling technique is used that removes hazardous substances such as BFRs. Further, we do recommend that white EPS that resemble insulation plates should be measured with XRF at sorting facilities in dubious cases, or simply not allowed to be recycled.

Integration of EPS sorting with XRF does have some potential to sort out more ambiguous EPS samples or complex EPS waste streams; which would be desirable to increase recycling rates. In this and a similar study by Harrad et al. (2019), no false negatives were accounted (bromine not found, but hazardous BFRs identified). Also, Sharkey et al. (2018) found that XRF analysis of EPS proved reliable as a "pass/fail" screening tool for LPCL compliance. Acting on pseudo-false positives (i.e. samples with measurable bromine, but no BFRs based on targeted analyses) by removing them from the recycling loop would only cause a small portion of EPS to not be recycled, which would also be favourable from a precautionary point of view. Therefore, XRF would be a recommended investment for other EPS streams than packaging waste, particularly for the sorting of large pieces that could contain BFRs, in order to increase EPS recycling targets while avoiding BFR contamination. It would also be recommended in regions where packaging EPS is already impacted by unintended BFR contamination through recycling.

The use of advanced bromine screening via acetone dissolution and targeted chemical analysis is likely not possible to be made at the level of individual EPS pieces at MWSFs in a cost-effective way. However, such techniques could play an important role for characterizing produced, recycled EPS, along-side target BFR and non-target analysis. Analysis of acetone-soluble and non-acetone soluble BFRs could help to monitor in what form bromine is appearing in recycled EPS.

5. International relevance

Data on EPS sorting practices in other countries is very hard to come by. Thus, in future studies on EPS recycling, or other plastic recycling, the context of sorting infrastructure and compliance should be a central feature when investigating how BFRs or other hazardous substances enter the circular economy. An important way forward is that construction EPS should be kept separated from packaging EPS for recycling, for separation of BFR and non-BFR waste. However, more investigations are needed in other countries to ensure that BFRs from e.g. construction EPS, do not enter the packaging EPS waste stream. The Korean (Rani et al., 2014), UK and Ireland (Abdallah et al., 2018) studies report higher concentrations of BFRs in packaging than observed here, which could have been due to mixed sorting into the recycling stream. Based on these discrepancies, the results in this study for Norway may not be applicable to EPS sorting and recycling schemes in other countries. XRF screening of visually sorted EPS packaging waste in other countries would be the most rapid way to compare with the Norwegian situation.

On an international level, and particularly in Europe, EPS recycling is increasing, as emphasised by a voluntary pledge submitted by the association "European Manufacturers of EPS" (EUMEPS) on behalf of its members to increase their recycling targets by 2025 (EUMEPS, 2018). This is a positive trend; however as part of this pledge, it should be ensured that as little BFRs end up in recycled EPS products as possible.

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.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.hazl.2021.100016.

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