Baltic Marine Environment Protection Commission

A3.2 Support for, and harmonisation of, regional work on Descriptor 10

Main report on microlitter

BLUES

Co-funded by the European Union

Activity 3- Marine litter

2023

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Start date and end date of the project
25/01/2021 – 24/01/2023

A3.2 Progress development of microlitter

Results summary on subtasks

In order to support the development of microlitter monitoring in 1) the water column and 2) seabed sediments five subtasks were conducted.

Subtask 3.2.1: Review / evaluation of current approaches applied for monitoring of microlitter in the water column and in seabed sediments

Within this subtask a survey on currently applied or planned monitoring strategies on microlitter in the water column and in seabed sediments was conducted reaching out to national experts from HELCOM countries. The survey covered information and data on general aspects such as the current status of the monitoring or whether monitoring data were already available. Furthermore, the monitoring strategy was asked for providing information on existing or intended number and location of microlitter monitoring stations and sampling strategy and methods and the protocols and methods applied in terms of sample treatment and analyses in the laboratory. Feedback was received from nine HELCOM countries on both compartments and the compiled outcome was presented and discussed within a technical workshop in June 2021 (HELCOM BLUES 3.2 WS 1-2021).

Subtask 3.2.2: Drafting guidelines for the monitoring of microlitter in the water column and in seabed sediments

Based on the outcomes of the method survey, draft guidelines were elaborated both for monitoring of microlitter in the water column and in seabed sediments. The guidelines were further discussed based on comments received from the HELCOM Expert Group on Marine Litter within two technical workshops (HELCOM BLUES WS 3.2-2022 in February and IC WS BLUES 3.2-2022 in September 2022) and amended accordingly. The draft guidelines were subsequently considered in the HELCOM framework and are included in the HELCOM Monitoring Guidelines catalogue and available in the HELCOM website (guidelines in the water column and in sediments, in addition, please see A3.2 Annex 1 and 2).

Subtask 3.2.3: Data collection from HELCOM countries on microlitter in the water column and in seabed sediments

A comprehensive literature survey and the consultation of the EMODnet database was conducted in order to gather existing data on microlitter in the water column and in seabed

sediments. For the water column, the existing data from literature was compiled as part of subtask 3.2.5 (screening study). As for seabed sediments, only few research data are available and the referring data are not comparable and not suited to be included for e.g. the calculation of baselines since they do not match the criteria that are now set within the guidelines.

Subtask 3.2.4: Specification of prerequisites for the monitoring of microlitter in the water column and in seabed sediments

The prerequisites for future monitoring were identified based on the parameters and criteria provided within the monitoring guidelines. Furthermore, major hindrances still existing were identified within a technical discussion with EG Marine Litter (December 2022). These hindrances and challenges mostly refer to management structures, logistic issues, resources and lacking research findings as for example on the spatial and temporal representativeness of the monitoring. Both prerequisites and identified hindrances are summarised within a document provided in A3.2 Annex 3.

Subtask 3.2.5: Screening studies on microlitter in the water column and in seabed sediments

Two screening studies were carried out: 1) a review of existing data from publications and available within EMODnet on microlitter in the water column which revealed that data are still scarce and not comparable (see overview provided in A3.2 Annex 4); and 2) a screening study for the validation of the guidelines on monitoring of microlitter in seabed sediments in the German Baltic Sea. In the latter, 14 monitoring stations in transects along major estuaries and additional three stations within the Exclusive Economic Zone were sampled and analysed according to the HELCOM guidelines. In general, the analyses revealed that the current guidelines are feasible and can be applied on real environmental samples. The results show a clear tendency of decreasing microlitter concentrations with increasing distance to the coastline. Furthermore, replicate analyses within stations and within samples provided good and reproducible findings lacking large variations. The outcomes of this screening study were presented at the Micro 2022 conference (see A3.2 Annex 5) and are envisaged for scientific publication.

Key messages for science and policy

Key messages for science

1) Further discussions are still needed especially in terms of the selection of monitoring stations, the precision on QA/QC measures within sample processing and data reporting.
2) There is still a lack of research findings concerning spatial and temporal representativeness of the monitoring strategy.

3) Data from the screening study on microlitter in seabed sediments indicate decreasing concentrations with increasing distance to the coastline and reproducibility of findings from replicate samples.

Key message for policy makers

1) Major hindrances for microlitter monitoring are mostly related to management structures, logistics and resources that are to be solved for a consistent HELCOM wide monitoring and the generation of baseline and threshold values.
2) Cooperation between countries in terms of monitoring sampling campaigns and laboratory analyses should be further evaluated.
3) Additional efforts are to be made to support scientific approaches to assess the representativeness of data.

Use of results

The results from activity 3.2 support:

- HELCOM, with the provision of new monitoring guidelines for microlitter monitoring in the water column and in seabed sediments.
- Implementation of the Baltic Sea Action Plan, in particular action HL32 on the agreement on core indicators and harmonized monitoring methods to evaluate quantities, composition, distribution and sources of marine litter, including microlitter.
- The EU Marine Strategy Framework Directive (MSFD), contributing the reporting on Descriptor 10 (Marine Litter) / D10C2 microlitter in water and sediments.
- Additional EU processes, with the provision and inclusion of HELCOM monitoring guidelines within TG Litter approaches towards the MSFD guidance chapter on microlitter (in preparation).

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Baltic Marine Environment Protection Commission

BLUES

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A3.2 Annex 1

HELCOM Guidelines on monitoring of microlitter in seabed sediments

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HELCOM Guidelines on monitoring of microlitter in seabed sediments in the Baltic Sea

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Content

Pre-Notes

In the context of the HELCOM BLUES project (DG Environment, MSFD, https://blues.helcom.fi/) a survey on existing and planned method approaches for the monitoring of microlitter in the Baltic Sea was conducted and compiled. This draft document on guidelines for sampling, sample treatment and analysis of microlitter within HELCOM is based on the outcomes of the discussions during three workshops with national experts on microlitter held on June 30, 2021, February 8, 2022 and September 6, 2022.

1. Introduction

Marine Litter and Microlitter are defined according to Commission Decision 2017/848 (2017) and UNEP (2022): "Marine litter is any persistent, manufactured or processed solid material discarded, disposed of or abandoned in the marine and coastal environment" (UNEP, 2022).

Marine microlitter is marine litter with a length of its maximum dimension below 5 mm.

The scope of microlitter monitoring within HELCOM is in accordance with MSFD Com Dec: D10C2: "micro-litter shall be monitored in the surface layer of the water column and in the seabed sediment and may additionally be monitored on the coastline. Micro-litter shall be monitored in a manner that can be related to point-sources for inputs (such as harbours, marinas, waste-water treatment plants, storm-water effluents), where feasible" (Commission Decision (EU) 2017/848, 2017).

2. Sampling of seabed sediments for microlitter monitoring

2.1 Sampling conditions

2.1.1 Number and location of monitoring stations

The number of monitoring stations surveyed by each country depends on the heterogeneity across stations and areas as well as on how many sub-basins each respective country encompasses. For shared sub-basins, there is a shared monitoring responsibility. The distribution of monitoring stations should represent variation within HELCOM sub-basins and should, where possible, integrate stations for target and measure monitoring[1] (i.e. near coast locations that are related to potential point-sources or locations of potential accumulation areas) as well as state monitoring[2] (i.e. open water or offshore-locations) according to the technical guidance on monitoring for the Marine Strategy Framework Directive (Zampoukas et al. 2014). It is further suggested to include preferably stations with known sediment deposition rates.

Where feasible, stations for monitoring of microlitter should correspond to existing monitoring stations from other monitoring programmes such as contaminants in sediments.

[1] "Target and measure monitoring (relating to Art. 10 and 13 MSFD) which compares to WFD operational monitoring: This requires additional monitoring (in terms of indicators/parameters, sampling frequency and stations) in those areas and for those ecosystem components for which GES has been failed and for those pressures, which are responsible for failing GES and for which environmental targets have been set. Monitoring should enable to assess progress towards GES and achieving targets and the efficiency of measures." (Zampoukas et al. 2014: 15).

[2] "State monitoring (relating to Art. 8, 9 MSFD) which compares to WFD surveillance monitoring: It aims at long-term monitoring and at surveillance monitoring for an overview of the state of the environment and is the backbone of MSFD monitoring. It is sufficient where GES is achieved for the individual ecosystem component. State monitoring includes the features, activities and pressures relevant for GES. It includes monitoring of additional parameters under Annex III MSFD to assess the extent and intensity of human activities and resulting pressures and their changes as well as changes in natural conditions." (Zampoukas et al. 2014: 15).

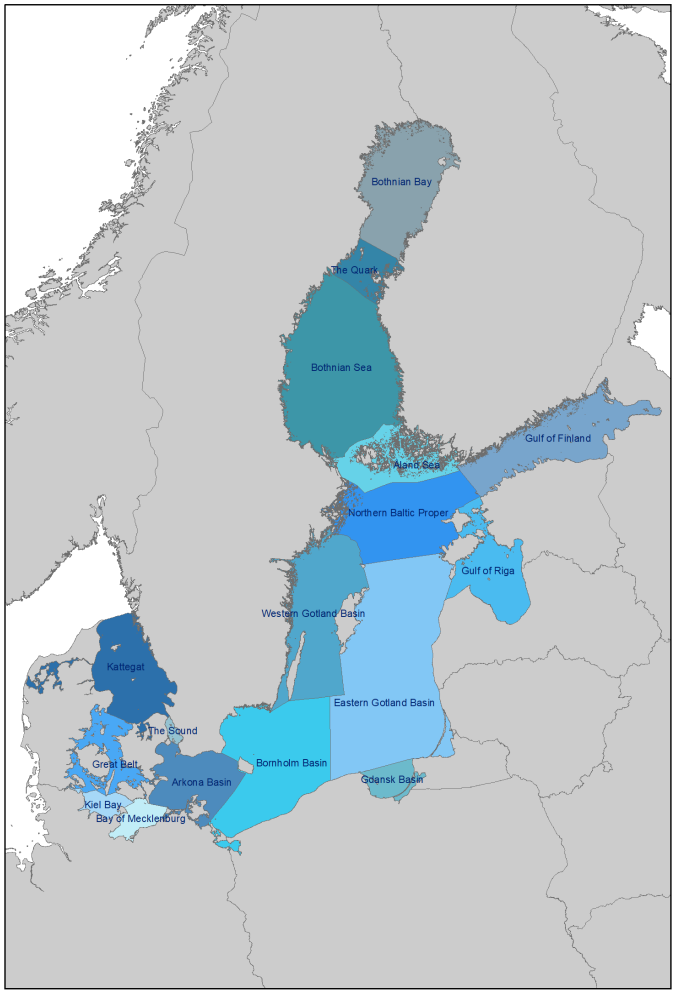

Figure 1. Map of the Baltic Sea presenting the HELCOM sub-division into 17 open sea sub-basins (HELCOM 2022).

2.1.2 Frequencies and time of sampling

The frequency of monitoring for microlitter in seabed sediments is still under discussion. It is suggested that monitoring frequency should be determined on the basis of further analysis e.g. on sampling methods, variance in microlitter concentrations and local conditions. It is further proposed to consider different frequencies in the case of parallel investigations at target and measure monitoring versus state monitoring stations.

The sampling time depends on feasibility and whether the sampling is carried out in accordance with other monitoring programmes that require a specific season for sampling.

2.2 Sampling techniques

2.2.1 Sampling device, sample volume, replicates

Sampling of seabed sediments can be done via grabs or corer-based approaches (e.g. Van Veen grab, box corer, Gemax corer, Kajak corer).

Samples are taken within the upper 2 to 5 cm layer of the sediment by means of stainless-steel equipment (spoons, trowels) and are transferred to pre-cleaned labelled glass or metal/aluminium jars. For monitoring stations with known sedimentation rate and absence of bioturbation processes

the sample depth may be adjusted in order to take into account the sedimentation of specific periods or assessment cycles.

The total sample volume relates to the sampling device. It is recommended to retrieve a minimum volume that allows for repetitions of the analyses and the determination of additional sediment-related parameters.

It is discussed to sample in duplicates or replicates (2-3 hauls) and/or to combine the resulting samples to one composite laboratory sample for further analyses. It is also discussed if replicates are only taken at state monitoring stations.

2.2.2 Recording of basic parameters, sampling protocol

Basic parameters during sampling shall be recorded and include:

a) Mandatory: date, station name and internal identification code (ID), coordinates, water depth, depth of sampled sediment surface layer.

b) Optional: weather and sea conditions, substrate, station classification.

Sample codes and parameters are documented in the sample documentation form. The respective sample containers are labelled with (at least): date, station code, station name and the internal code for laboratory processing (ID).

2.2.3 Sample transportation, preservation and storage

Samples are stored in glass, aluminium or metal containers providing light-absence and low temperatures. The use of plastic bags or containers is to be avoided. The storing conditions depend on the storage time and conditions during the sampling campaign and/or on the schedule of the laboratory conditions.

2.3 Sampling QA/QC

To minimise background contamination, the following measures should be considered within the sampling campaigns, also when they are carried out in parallel to other monitoring campaigns:

c) Use of glass or aluminium/metal materials only, avoid the use of synthetic materials.

d) Pre-cleaning of sample containers and instruments with filtered water and/or ethanol or isopropanol. Glassware can also be subject to baking within a muffle oven at 500 °C.

e) Operators to avoid wearing synthetic clothes . Operators to position facing the wind while retrieving the sample. Operators to take care that potential contamination sources during sampling and sample processing are avoided.

f) Integration of blank samples: a representative number of blank samples are integrated to account for contamination during sampling. The number of blank samples should be at least 3. The total number of blank samples should be representative for varying sampling conditions during the respective sampling campaign and thus, should reflect the specific contamination potential e.g. through varying weather conditions, varying operators wearing varying clothes. It is recommended to retrieve material from any device of synthetic polymer origin implemented during sampling. These comparative materials should be investigated for their polymer composition to enable exclusion of clearly identified contamination from sampling devices.

A proportion of 10 % blank samples of the total number of sediment samples is recommended for homogeneous conditions during the sampling campaign.

For generating blank samples, an empty sampling container is positioned next to the sample and opened while retrieving the sample. The resulting blank sample is subject to laboratory analyses in the same manner as sediment samples.

3. Sample treatment / laboratory analyses

Sample treatment and laboratory analyses can be done applying different methods when specific quality criteria are ensured. Any sample treatment needs to ensure not to harm synthetic polymers by applying strong chemicals and high temperatures. The treatment process and methods applied need to be controlled via contamination control and recovery tests with reference samples.

3.1 Laboratory QA/QC

3.1.1 Contamination control

Appropriate measures to reduce air contamination, cross-contamination and contamination control must be taken during laboratory analysis. These include:

- Wearing of personal protection equipment made of natural materials (cotton laboratory coats, avoid plastic fibre face masks).
- Ensuring clean laboratory conditions (regular cleaning, regulated air circulation, minimized presence of staff, use of clean room and laminar flow chambers combined with fume hoods if possible).
- Avoidance of any plastic materials during analyses (preferred use of glass and stainless steel materials).
- Pre-filtration of water and chemical solutions with filter pore size significantly lower than minimal cut-off size of targeted particles in the samples.
- Pre-cleaning of beakers and instruments.
- Pre-cleaning of filters (rinsing, annealing according to filter material).
- Covering samples and working solutions throughout the sample processing.
- Reduction of processing steps as far as possible.
- Inclusion of a relevant number of blank samples analysed in parallel with each sample series.
- Inclusion of a relevant number of reference samples analysed in parallel with each sample series.

3.1.2 Blank samples and recovery tests (mandatory)

g) A relevant number of blank samples is to be analysed in parallel with each sample series (set of samples investigated in parallel in one laboratory processing cycle). Combining field blank and laboratory blank samples is not recommended since the number of samples processed within one sample series may differ from the number of samples being representative for the respective field blank sample.

h) Microlitter particles detected within both, field and laboratory blank samples, are used to calculate the limit of detection (LOD - mean + 3 x standard deviation of the particle concentration) according to McDougall et al. (1980). LOD thus reflects the efficiency of the precautionary methods during sampling and sample processing of the respective laboratory. LOD is reported within the data to EMODnet. Blank values are not subtracted from the results on sediment samples.

i) A relevant number of reference samples is to be analysed in parallel with each sample series. Reference samples reflect the efficiency of the respective laboratory protocol and are treated in the same manner and throughout all steps as the sediment samples.

j) Reference samples should encompass real sediment samples that are spiked with a relevant number of synthetic polymer particles that are representative for dominating size categories, morphologies and polymer composition of the particles to be detected within the sediment samples. The number of added reference particles is to be discussed. The number of reference particles will affect the resolution of the recovery

rates, thus, a number of at least 50 reference particles for both fragments and fibres could be recommended leading to a resolution of 2 %.

k) The recovery ratio (%) is calculated for re-detected added reference particles as the mean value accounting for different size categories, morphologies and polymer composition. It is recommended to include reference material containing three types of polymer with different densities, three morphologies and a similar size to the targeted lower cut-off size (i.e. 100 µm) of particles according to Cui et al. (2022). The mean recovery ratio is reported together with the data to EMODnet. Results on sediment samples are not corrected for recovery rates.

3.2 Sample volume and weight for laboratory analyses

In general, the sample volume for laboratory analyses is dependent on sample composition, sample storage conditions and further sample processing methods.

Field samples are homogenised by stirring with glass or metal spatulas or spoons. The respective volume for laboratory analyses is determined (e.g. by using a metal measuring spoon) and weighed to a pre-cleaned beaker with an analytical balance (accuracy minimum 0.01 mg).

In parallel, a second aliquot of the field sample is investigated for water content in order to determine the dry weight of sediment. Therefore, an aliquot of approximately 10 ml is transferred and weighed into an evaporating dish and dried at 105 °C. Following cooling to room temperature within a desiccator, the samples are reweighed, and dry weight is calculated. Equally, dry weight of sediment can be calculated from weight difference derived through freeze drying.

3.3 Sample digestion

In general, the order of digestion and density separation depends on the sample treatment protocol and particle analysis technique of the processing laboratory.

Optional digestion protocols cover oxidative, enzymatic, alkaline or mixed treatments. The implementation of acid digestion is not recommended since especially strong acids proofed to affect synthetic polymers. The duration of the sample digestion depends on the selected digestion protocol.

The application of low temperatures and stirring of the samples are optional add-ons within sample digestion. The application of temperatures >40°C (>50°C if enzymatic digestion is applied) is to be avoided since it may damage synthetic polymers.

After digestion, the digestion solution is rinsed-off over a sieve with the mesh size of the minimum size of targeted particles (100 µm mandatory, see chapter 3.5 and 4.3 for further options).

If particle dimensions are not determined by single particle a size separation step with a sieving cascade encompassing at least 100, 300 and 1000 µm can be applied at this stage (smaller mesh sizes are optional). If pre-sieving at the beginning of the laboratory processing has not been applied, an additional sieve with a mesh size of 5000 µm can be integrated at this stage.

3.4 Density separation

The choice of the density solution and the device used for density separation depends on the respective protocol applied. Density solutions cover zinc chloride ($ZnCl_2$), sodium iodide (NaI), and sodium polytungstate ($NaWO_4$) with a minimum density of 1.5 g/cm³. The application of solutions with densities >1.7 g/cm³ is recommended since this will distinctly improve the recovery rates of synthetic particles of higher material densities. The use of sodium chloride (NaCl) is not recommended since a relevant number of synthetic polymers will not be recovered due to low solution density.

In general, samples are introduced into the density separation solution, stirred for 10 minutes and left for settling for 24 h. The supernatant suspension is rinsed thoroughly with filtered water and

transferred onto filters applicable for the further particle identification. Filters are left to dry in pre-cleaned glass petri dishes. It is recommended to repeat the density separation process at least once.

3.5 Particle identification

The identification of synthetic particles depends on the device available and varies between optical microscopic identification, spectroscopic approaches like FTIR and Raman spectroscopy and staining approaches like Nile red staining in combination with fluorescence microscopy. Particles are identified according to numbers, size classes, morphology, colours (optional) and polymer composition (on at least a subset). The minimum cut-off size for data to be reported is 100 µm (see 4.3 for size classes and options).

3.6 Polymer identification

The determination of at least a subset of particles for their polymer composition via FTIR or Raman spectroscopy is mandatory. Device settings and minimum library match (%) attributed is to be recorded within the metadata to EMODnet. Spectra libraries utilised for polymer composition determination should integrate spectra from synthetic and organic components and weathered synthetic polymers. It is suggested to agree on one or several libraries that are used by all processing laboratories and/or to generate a combined FTIR and/or Raman spectra library for HELCOM microlitter monitoring.

It is recommended to analyse the polymer composition on a representative subset with a minimum of 10 % (preferably at least 20 particles per sample) of synthetic particles identified within the size categories from 100 to 1000 µm. The subset size of particles identified in any smaller size category is to be discussed. The particles integrated in the subset are to be selected representatively according to size categories and morphologies.

4. Parameter and data recording

Parameters are to be recorded according to EMODnet requirements (see section 5). Data can also be reported to ICES DOME when parameters and attributes and e.g. harvesting of data from EMODnet will be harmonised (and the consent of the country is given). The reporting to or harvesting of data through ICES DOME is under discussion.

Parameters to be recorded encompass the following:

4.1 Numbers

The recording of number of particles identified as synthetic polymers/microlitter is mandatory.

Data are calculated to number of particles and optionally weight in grams per kg dry weight of sediment. At this stage, no recommendation on re-calculating number of particles into mass is given. The development of conversion algorithms based on polymer composition and particle size/volume is to be evaluated.

4.2 Morphology

The morphology of all identified particles is to be recorded according to the following morphology classes:

Table 1 Morphology classes to be used to report all identified particles.

EMODnet identifier "micro-litter morphology"	Name	Definition according to EMODnet	Definition according to GESAMP 2019 (Kershaw et al. 2019)
H0100004	Filaments	Slender thread-like micro-litter particles	"Line" (Fibre, filament, strand): long fibrous material that has a length substantially longer than its width
H0100005	Films	Micro-litter particles derived from plastic sheets or thin plastic films	"Film" (sheet): flat, flexible particle with smooth or angular edges
H0100006	Foams	Any kind of micro-litter particle made of plastic foam, including styrofoam	"Foam" (EPS, PUR): near spherical or granular particle, which deforms readily under pressure and can be partly elastic, depending on weathering state
H0100002	Fragments	Irregularly-shaped plastic micro-litter particles with broken off edges that may be rounded or angular	"Fragment" (granule, flake): irregular shaped hard particles having appearance of being broken down from a larger piece of litter
H0100003	Pellets	Micro-litter particles from industrial origin only. In comparison with granules, pellets are usually flat on one side, rough surface and irregular, round shapes	"Pellet" (resin bead, Mermaids tears): hard particle with spherical, smooth or granular shape
H0100009	Granules	Micro-litter particles with smooth spherical shape. In comparison with pellets, they have a rounder shape	

It is to be evaluated whether microbeads are to be reported as a single class or identified from the data set as morphology: granules and the (smaller) dimension in size compared to pre-production resin pellets.

It is to be evaluated whether "pellets" and "granules" should be separate classes. In addition, it has to be considered that "film" and "foam" might not be identified due to restrictions of devices or protocols especially within the smaller size fractions.

4.3 Particle dimensions

The dimensions of identified particles should be recorded according to the following size classes:

- 100 – 299 µm
- 300 – 999 µm

- 1000 – 4999 µm

The reporting of size classes below 100 µm is optional according to the following size classes:

- 50 – 99 µm
- 20 – 49 µm
- <20 µm

It is to be pointed out that results may be biased if particle dimensions are retrieved from mesh sizes from sieving and filtering or measuring of actual particle length and width dimensions.

The reporting of absolute dimensions on particle length and/or particle width is optional.

Sizes of particles are defined according to:

l) Length (maximum Ferret diameter in longitudinal orientation)

m) Width (maximum Ferret diameter perpendicular to the identified length transect)

Fibres with a length > 5000 µm are considered "mesolitter" and are therefore excluded from the data analysis.

4.4 Polymer composition

Polymer composition is to be reported according to polymer classes and is to be defined for at least a subset of identified synthetic particles.

It is suggested to align the polymer types according to the list modified from AMAP 2021 (see Table 2) but to set up a short list with prioritised synthetic polymers that are predominantly found in environmental samples and that at least have to be reported when occurring.

Table 2 Polymer types for data reporting (modified from AMAP 2021: 225)

Polymer type name	**Examples of materials included (detailed level)**	**Modifications compared to AMAP (2021)**
Acrylonitrile based	e.g. acrylonitrile butadiene styrene (ABS), polyacrylnitrile (PAN)	Modified to "Acrylonitrile based", PAN removed from polymer type and integrated here as an example
Cellulose based	e.g. cellulose acetate (CA), cellulose nitrate (CN)	Modified to "cellulose based", examples added
Polyamide based	e.g. all types of polyamide (PA) like various nylons	
Polycarbonate based	e.g. polycarbonate (PC)	Modified to "polycarbonate based"
Polychlorinated polymers	e.g. polyvinyl chloride (PVC), chlorinated PE, various chlorinated polymers	
Polyester based	e.g. polyethylene terephthalate (PET), all other types of polyesters	Modified to "polyester based"
Polyethylene based	e.g. high density polyethylene (HDPE), low density polyethylene (LDPE), and copolymers with a major PE fraction including ethylene-vinyl acetate copolymer (EVA)	EVA removed from polymer type and integrated into polyethylene based.
Polyfluorinated polymers	e.g. polytetrafluoroethylene (PTFE)	
Polymeth(ester)acrylate based	e.g. all types of polymeth(ester)acrylate (PM(ester)A)	
Polypropylene based	e.g. polypropylene (PP), and copolymers with a major PP fraction	
Polystyrene based	e.g. polystyrene (PS), and copolymers with a major PS fraction	

Polyurethane based	e.g. all types of polyurethane (PUR)	
Rubbers, automotive	e.g. styrene butadiene rubber (SBR), tire wear	SBR added as an example
Varnish/paint particles	If different from PM(ester)A	
Other plastics	e.g.polyether ether ketone (PEEK), polyoxymethylene (POM), polyvinyl acetate (PVA), polylactic acid (PLA), polyhydroxyalkanoate (PHA)	Examples added / moved from single polymer classes
Other rubbers	e.g. ethylene propylene diene monomer rubber (EPDM), nitrile rubbers, natural rubbers, silicone	Examples added / moved from single polymer classes / rubber types (refers to "rubbers sealing", "nitrile rubbers", "natural rubbers and derivates", "silicone rubbers and derivates"
Other microlitter materials	e.g. metal, glass	Examples added
Other semi-synthetic polymers	e.g. rayon	Polymer type added / introduced

4.5 Optional parameters

The recording of particle colours and/or transparency is optional. Colours and transparency are classified according to EMODnet:

Colour classes:

- black / grey
- blue / green
- brown / tan
- white / cream
- yellow
- orange / pink / red
- purple
- multicolour

It is suggested and discussed to include a class „colourless" in order to address particles derived from colourless and transparent foils or particles from e.g. (uncoloured) plastic bottles.

Transparency:

- yes
- no

4.6 Sediment parameters

Mandatory parameters: dry weight of sediment (g, weight after drying at 105°C, according to ISO 11465:1993 (2020).

Optional parameters: water content (%, weight difference between 40°C and 105 °C according to DIN ISO 11465), carbonate content (%), total organic carbon (%).

It is under discussion whether organic content (%, to be determined by loss on ignition at 550 °C) and grain size distribution according to sand (63-2000 µm, %) and clay + silt (2-63 µm, %) should be mandatory or optional parameters.

5. Data reporting to EMODnet

Data are to be reported to EMODnet according to current specifications provided by EMODnet (i.e. Vinci et al. 2021).

The reporting to or harvesting of data through ICES DOME is under discussion.

The following lists comprise parameters (mandatory and optional), EMODnet codes and descriptions where available and suggestions for modifications or the integration of further parameters following the discussions and suggestions provided within these draft guidelines and first evaluations through EMODnet.

Parameters and related attributes are under continuous development. Therefore, it is recommended to consult the latest tables and vocabularies online at the NERC Vocabulary Server (NVS).

Table 3 Current list of default (green), mandatory (orange) and optional (light orange) parameters to be reported (modified from Vinci et al., 2021, p7).

Label/column header	Concept id	Use	Comments
Cruise		metadata/mandatory (ODV Default)	
Station		metadata/mandatory (ODV Default)	
Type		metadata/mandatory (ODV Default)	The suggestion is to use type "B". From manual: 'B' for bottle profile data. For time series and trajectories set to 'B' for small (<250) row groups
YYYY-MM-DDThh:mm:ss.sss		metadata/mandatory (ODV Default)	Start date/time. Format must be adapted to the date value (for example YYYY-MMDDThh:mm is second are not available)
Longitude [degrees_east]		metadata/mandatory (ODV Default)	start point coordinates
Latitude [degrees_north]		metadata/mandatory (ODV Default)	start point coordinates
LOCAL_CDI_ID		metadata/mandatory (ODV Default)	
EDMO_code		metadata/mandatory (ODV Default)	EDMO_CODE of the data centre distributing the data (the one connected to the CDI service)
Bot. Depth [m]		metadata/mandatory (ODV Default)	Field empty if no data
MinimumObservation Depth [m]	MINWDIST	mandatory in ODV micro-litter	
MaximumObservation Depth [m]	MAXWDIST	mandatory in ODV micro-litter	
SampleID:INDEXED_TEXT	SAMPID01	mandatory in ODV micro-litter	
Microlitter_Type:IND EXED_TEXT	SDN:H01	mandatory in ODV micro-litter	Type of the item (H01 SDN vocabulary); MLITYPS
Microlitter_Size:INDE XED_TEXT	SDN:H03	mandatory in ODV micro-litter	Size classes (H03 SDN vocabulary), MLITSZS

Microlitter_Count [Dimensionless]	MLITCNTS	mandatory in ODV micro-litter	Number of items collected. It's the official mandate from MSFD to provide the count of collected microplastics.
EventEndDateTime [YYYY-MMDDThh:mm:ss.sss]	ENDX8601	additional/optional	End date/time
EventEndLongitude [degrees_east]	ENDXXLON	additional/optional	End point coordinates. Either End Lat/Lon or SamplingEffort are mandatory
EventEndLatitude [degrees_north]	ENDXXLAT	additional/optional	End point coordinates. Either End Lat/Lon or distance are mandatory.
Microlitter length	NEW	additional/optional	
Microlitter width	NEW	additional/optional	
Microlitter_Weight [g]	MLDWSD01	additional/optional	Weight of the collected items, not mandatory Information in grams
Microlitter_Color:IN DEXED_TEXT	MLITCOLS	additional/optional	Colour classes (H04 SDN vocabulary)
Microlitter_Transparency:INDEXED_TEXT	MLITROPS	additional/optional	Transparency classes (H06 SDN vocabulary)
Microlitter_Polymer_type:INDEXED_TEXT	MLITPOLS	additional/optional	Polymer type of the micro-litter (H05 SDN vocabulary)
WMO_Sea_State [Dimensionless]	WMOCSSXX	additional/optional	Sea conditions following the Douglas scale
Wind_direction [degT]	EWDAZZ01	additional/optional	Direction relative to true north from which the wind is blowing
Wind_speed [m/s]	WSBZZ01	additional/optional	Sustained speed of the wind (distance moved per unit time by a parcel of air) parallel to the ground at a given place and time.
Sampling_protocol	SAMPPROT	additional/optional	The name of, reference to, or description of the method or protocol used to produce the sample

6. References

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Annex

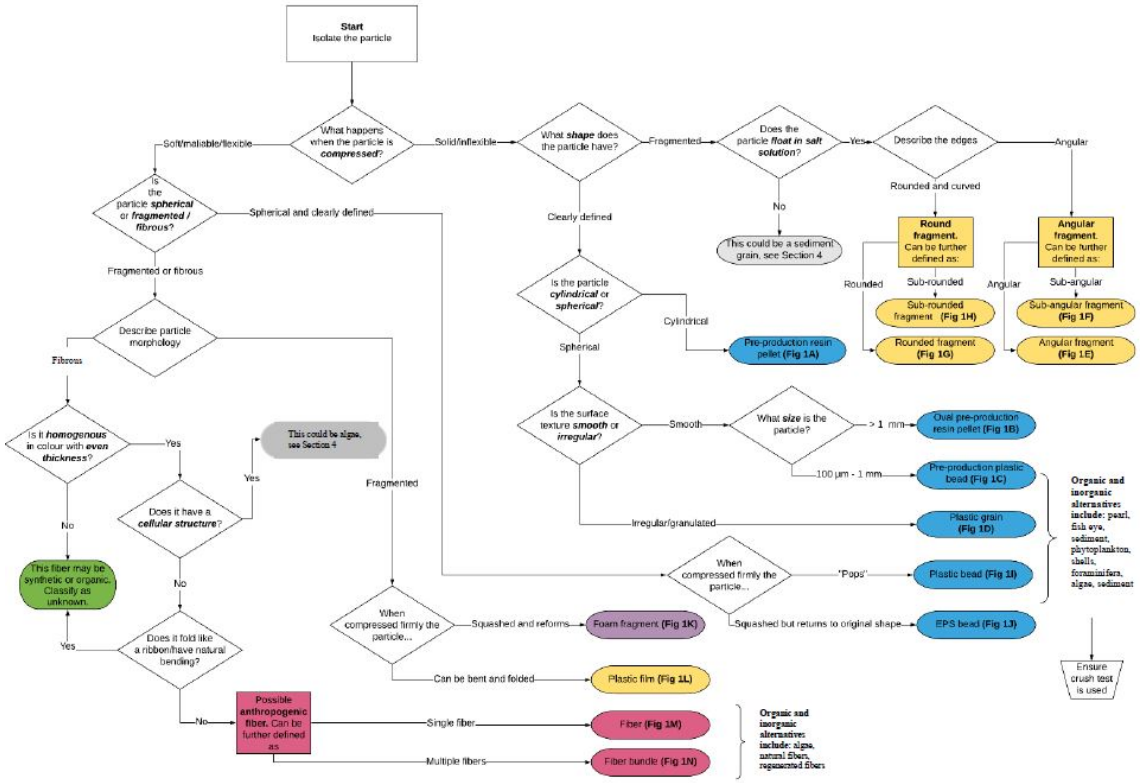

Figure A1 Proposed flow chart for the visual identification of microplastics. (AMAP 2021: 223, reproduced from Lusher et al., 2020).

Baltic Marine Environment Protection Commission

BLUES

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A3.2 Annex 2

HELCOM Guidelines on monitoring of microlitter in the water column in the Baltic Sea

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Contents

Pre-notes

In the context of the HELCOM BLUES project (DG Environment, MSFD, https://blues.helcom.fi/) a survey on existing and planned method approaches for the monitoring of microlitter in the Baltic Sea was conducted and compiled. This draft document on guidelines for sampling, sample treatment and analysis of microlitter within HELCOM BLUES project is based on the outcomes of the discussions during three workshops with national experts on microlitter held on 30 June 2021, 8 February 2022 and 6 September 2022.

1. Introduction

Marine Litter and Microlitter are defined according to Commission Decision 2017/848 (2017) and UNEP, 2022: "Marine litter is any persistent, manufactured or processed solid material discarded, disposed of or abandoned in the marine and coastal environment" (UNEP, 2022).

Marine microlitter is marine litter with a length of its maximum dimension below 5 mm.

The scope of microlitter monitoring within HELCOM is in accordance with MSFD Com Dec: D10C2: "micro-litter shall be monitored in the surface layer of the water column and in the seabed sediment and may additionally be monitored on the coastline. Micro-litter shall be monitored in a manner that can be related to point-sources for inputs (such as harbours, marinas, waste-water treatment plants, storm-water effluents), where feasible" (Commission Decision (EU) 2017/848, 2017).

2. Sampling of marine water column for microlitter monitoring

2.1 Sampling conditions

2.1.1 Number and location of monitoring stations

The number of monitoring stations surveyed by each country depends on the size of the area under responsibility of the respective country, as well as on how many sub-basins the country encompasses. For shared sub-basins, there is a shared monitoring responsibility. The distribution of monitoring stations should represent variation within HELCOM sub-basins (Figure 1) and should, where possible, integrate stations for target and measure monitoring[1] (i.e. near coast locations that are related to potential point-sources or locations of potential accumulation areas) as well as state monitoring[2] (i.e. open water or offshore-locations) according to the technical guidance on monitoring for the Marine Strategy Framework Directive (Zampoukas et al. 2014).

Where feasible, stations for monitoring of microlitter should correspond to existing monitoring stations from other monitoring programmes such as hydrochemical, hydrophysical and hydrobiological monitoring.

[1] "Target and measure monitoring (relating to Art. 10 and 13 MSFD) which compares to WFD operational monitoring: This requires additional monitoring (in terms of indicators/parameters, sampling frequency and stations) in those areas and for those ecosystem components for which GES has been failed and for those pressures, which are responsible for failing GES and for which environmental targets have been set. Monitoring should enable to assess progress towards GES and achieving targets and the efficiency of measures." (Zampoukas et al. 2014: 15).

[2] "State monitoring (relating to Art. 8, 9 MSFD) which compares to WFD surveillance monitoring: It aims at long-term monitoring and at surveillance monitoring for an overview of the state of the environment and is the backbone of MSFD monitoring. It is sufficient where GES is achieved for the individual ecosystem component. State monitoring includes the features, activities and pressures relevant for GES. It includes monitoring of additional parameters under Annex III MSFD to assess the extent and intensity of human activities and resulting pressures and their changes as well as changes in natural conditions." (Zampoukas et al. 2014: 15).

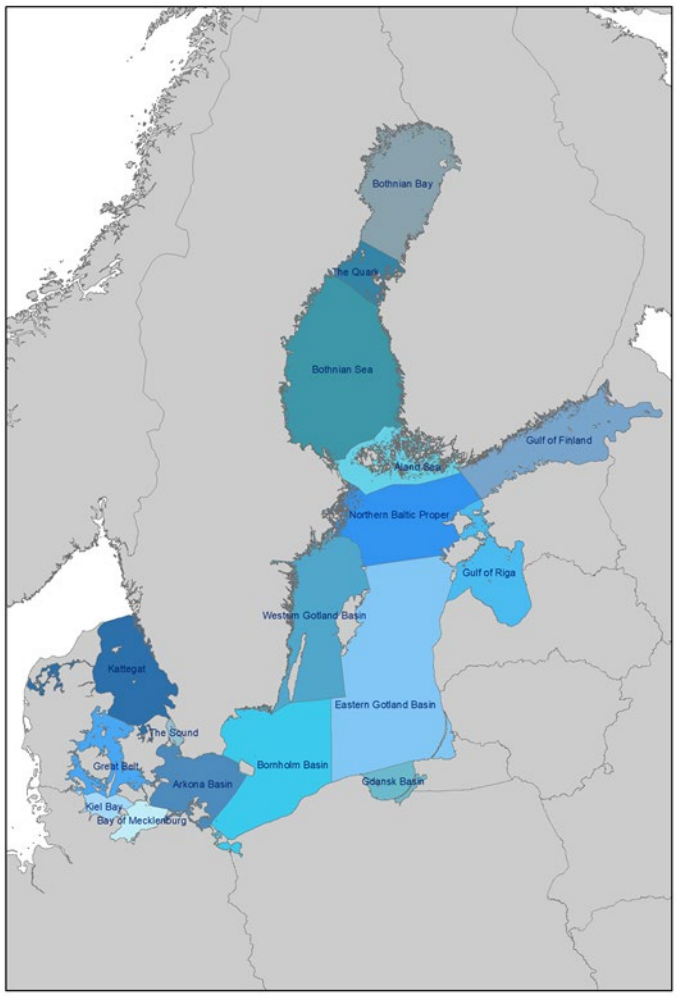

Figure 1: Map of the Baltic Sea presenting the HELCOM sub-division into 17 open sea sub-basins (HELCOM 2022).

2.1.2 Frequencies and time of sampling

The frequency of monitoring for microlitter in the surface layer of the water column is still under discussion. It is suggested that monitoring frequency should be determined on the basis of further analysis e.g. on sampling methods, variance in microlitter concentrations and local conditions. It is further proposed to consider different frequencies in the case of parallel investigations at target and measure monitoring versus state monitoring stations.

The sampling time depends on feasibility and whether the sampling is carried out in accordance with other monitoring programmes that require a specific season for sampling.

It should be taken into account that different weather events and hydrochemical, hydrobiological and hydrophysical peculiarities can influence microlitter distribution in the water column (e.g. downward mixing of microplastics from the sea surface due to a wind event or greater microplastic and algae concentrations because of a calm weather event). Seawater sampling during intense algal blooms or during the evening when zooplankton migrates to the surface should be avoided due to the fact that sample preparation in that case could become time and cost consuming.

2.2 Sampling techniques

2.2.1 Sampling device, sample volume, replicates, on-board sample processing

Sampling of microplastics from the water column can be done using nets (manta and plankton ones) and pumping systems with the mesh size of maximum 300 µm (optionally smaller mesh size can be used).

The depth of the sampled water column layer (surface of water column -up to 0. 25 m- or water column - > 0.25 m) should be registered. Sampling should not be impacted by the water mixing or particles created by the sampling vessel, therefore sampling devices should be positioned at the sides or stern of the vessel. For estimation of filtered water, the use of a volume flow meter is recommended, alternatively (only for nets) calculation of filtered water volume can be applied.

Filtered water volume is variable and dependent on water conditions (i.e. algae bloom) but should be at least 100 m^3 when using sampling devices with mesh size 300 µm, and at least 10 m^3 when using sampling devices with mesh size 100 µm. In cases when the requested sample volume cannot be conducted, sub-sampling for collection of needed sample volume is recommended. If a smaller mesh size or pumping system is used, it is acceptable for sample volume to be lower (due to potential mesh clogging or unrealistic pumping duration). It is supported to use manta trawl without replicates.

If manta net is to be used, it is to be noted that the speed of the vessel should not be higher than 2 knots in order to avoid clogging of the net.

After collection, samples should be concentrated using a sieve with a mesh size smaller than the sampling device mesh size, transferred to a pre-cleaned labelled glass tray and covered with a lid.

2.2.2 Recording of basic parameters, sampling protocol

Basic parameters during sampling shall be recorded and include:

- Mandatory: station name and sample ID (identification code), date, start and end (if applicable) coordinates, sampling device used, mesh size and opening (if applicable) of sampling device, depth of sampled water layer, filtered water volume, transect length and area (if applicable), water depth. Labelling the respective sample containers with (at least): station name and internal code for laboratory processing.
- Optional: weather and sea conditions (wind speed and direction, wave height and direction), station classification (coastal/offshore) and/or distance from the shore, amount of suspended solids (if applicable), CTD profile (if applicable).

Sample codes and parameters are documented in the sample documentation form.

2.2.3 Sample storage and preservation.

Samples should be stored in glass or metal containers, avoiding plastic ware as much as possible. It is recommended to store samples in low temperature (frozen or at maximum temperature of 2-6 °C) to stop biological processes. Alternatively, a conservation additive might be used.

2.3 Sampling QA/QC

To minimise background contamination, the following measures should be considered within the sampling campaigns, also when they are carried out in parallel to other monitoring campaigns:

- Use of glass and/or metal materials where possible, avoid the use of synthetic materials.
- Washing and rinsing of sampling devices before sampling to avoid cross-contamination.
- Pre-cleaning of sample containers and instruments with filtered water (mesh size smaller than lowest particle detection limit) and/or ethanol or isopropanol. Glassware can also be subjected to baking within a muffle oven at 500 °C.
- Operators to take care that potential contamination sources during sampling and sample processing are avoided (e.g. fleece sweaters hanging in the ship's laboratory). Wearing

brightly coloured work clothes for easy operator-generated contamination detection in samples. Operators to position facing the wind while retrieving the sample.

- Integration of blank samples: a representative number of blank samples should be integrated to account for contamination during sampling. The number of blank samples should be at least 3. The total number of blank samples should be representative for varying sampling conditions and thus, should reflect the specific contamination potential e.g. through varying weather conditions, varying operators wearing varying clothes.

  A proportion of 10 % blank samples of the total number of water microlitter samples is recommended for homogeneous conditions during the sampling campaign.

  For generation of blank samples, an empty sampling vessel is positioned next to the sample and opened while retrieving the sample. A field blank can also include a filtered distilled water rinse of a net. The resulting blank sample is subject to laboratory analyses in the same manner as water column microlitter samples.

  It is recommended to retrieve material from any device of synthetic polymer origin implemented during sampling. These comparative materials should be investigated for their polymer composition to enable exclusion of clearly identified contamination from sampling devices.

3. Sample treatment / laboratory analysis

Sample treatment and laboratory analysis can be done applying different methods when specific quality criteria are ensured. Any sample treatment needs to ensure not to harm synthetic polymers by applying strong chemicals and high temperatures. The treatment processes and methods applied need to be controlled via contamination control and recovery tests with reference samples.

3.1 Laboratory QA/QC

3.1.1 Contamination control

Appropriate measures to reduce air contamination, cross-contamination and contamination control must be taken during laboratory analysis. These include:

- Wearing of personal protection equipment made of natural materials (cotton laboratory coats, avoid plastic fibre face masks).
- Ensuring clean laboratory conditions (regular cleaning, regulated air circulation, minimized presence of staff, use of clean room and laminar flow chambers combined with fume hoods if possible).
- Avoidance of any plastic materials during samples processing (preferred use of glass and stainless-steel materials).
- Pre-filtration of water and chemical solutions with filter pore size significantly lower than minimal cut-off size of targeted particles in the samples.
- Pre-cleaning of filters, beakers and instruments by thoroughly rinsing with filtered (mesh size smaller than lowest particle detection limit) distilled water and/or ethanol or isopropanol or baking within a muffle oven at 500 °C.
- Covering samples and working solutions throughout the sample processing.
- Reduction of processing steps as far as possible.
- Inclusion of a relevant number of blank samples analysed in parallel with each sample series.
- Inclusion of a relevant number of reference samples analysed in parallel with each sample series to identify recovery rate is suggested.

3.1.2 Blank samples and recovery tests (mandatory)

- A relevant number of blank samples is to be analysed in parallel with each sample series (set of samples treated in parallel in one laboratory processing cycle). Combining field blank and laboratory blank samples is not recommended since the number of samples processed within one sample series may differ from the number of samples being representative for the respective field blank sample.
- Microlitter particles detected within both, field and laboratory blank samples, are used to calculate the limit of detection (LOD - mean + 3 x standard deviation of the particle concentration) according to McDougall et al. (1980). LOD thus reflects the efficiency of the precautionary methods during sampling and sample processing of the respective laboratory. LOD is reported within the data to EMODnet. Blank values are not subtracted from the results on water column microlitter samples.
- A relevant number of reference samples is to be analysed in parallel with each sample series. Reference samples reflect the efficiency of the respective laboratory protocol and are treated in the same manner and throughout all steps as the water microlitter samples.
- Reference samples should encompass samples that are spiked with a relevant number of synthetic polymer particles that are representative for dominating size categories, morphologies and polymer composition of the particles to be detected within the water samples. The number of reference particles will affect the resolution of the recovery rates, thus, a number of at least 50 reference particles for both fragments and fibres could be recommended leading to a resolution of 2 %.
- The recovery ratio (%) is calculated for re-detected added reference particles as mean value accounting for different size categories, morphologies and polymer composition. It is recommended to include reference material containing three types of polymer with different densities, three morphologies and a similar size to the targeted lower cut-off size (i.e. 100 µm) of particles according to Cui et al. (2022). The mean recovery ratios is reported together with the data to EMODnet. Results on water samples are not corrected for recovery rates.

3.2 Sample volume, sample preparation

Sample volume for laboratory analysis is dependent on the state of the sample, sampling conditions and further sample processing methods. In cases when the concentrated sample volume is high or the sample is rich on organic material, the splitting of the sample is supported, but it is recommended that sub-samples are treated proportionally, and that the total treated amount is not lower than the minimum recommended filtered water volume (see section 2.2.1.).

3.3 Sample digestion

In general, the order of digestion and application of density separation depends on the sample treatment protocol and particle analysis technique of the processing laboratory.

Optional digestion protocols cover oxidative, enzymatic, alkaline or mixed treatments. The implementation of acid digestion is not recommended since especially strong acids proved to affect synthetic polymers. The duration of the sample digestion depends on the selected digestion protocol and the complexity of the sample. It is recommended to use as little treatment steps as possible to avoid unintentional loss of particles.

The application of low temperature and stirring of the samples are an optional add-ons within sample digestion. The application of temperatures >40°C (>50°C if enzymatic digestion is applied) is to be avoided since it may damage synthetic polymers.

After digestion, the digestion solution is rinsed-off over a sieve or through a filter with a mesh size smaller than the minimum size of targeted particles.

If particle dimensions are not determined by single particle, a size separation step with a sieving cascade encompassing at least 300 and 1000 µm can be applied at this stage (smaller mesh sizes are optional). If pre-sieving at the beginning of the laboratory processing has not been applied, an additional sieve with a mesh size of 5000 µm can be integrated at this stage.

3.4 Density separation

Density separation might be applied in cases where inorganic material is present in the sample and may interfere with analysis.

The choice of the density solution and the device used for density separation depends on the respective protocol applied. Density solutions cover zinc chloride ($ZnCl_2$), sodium iodide (NaI) and sodium polytungstate ($NaWO_4$) with a mandatory minimum density of 1.5 g/cm³. The application of solutions with densities of >1.7 g/cm³ is recommended since this will distinctly improve the recovery rates of synthetic particles of higher material densities. The use of sodium chloride (NaCl) is not recommended since a relevant number of synthetic polymers will not be recovered due to low solution density.

In general, samples are introduced into the density separation solution, stirred for 10 minutes and left for settling for 24 h. The supernatant suspension is then transferred to filters, rinsed thoroughly with warm (<40 °C) filtered (mesh size smaller than lowest particle detection limit) water (additionally 50% ethanol can also be used) and saved for further particle treatment or identification. Filters are left to dry in pre-cleaned glass petri dishes.

3.5 Particle identification

The identification of synthetic particles depends on the availability of equipment and varies between optical microscopic identification, spectroscopic approaches like FTIR and Raman spectroscopy and staining approaches like Nile red staining in combination with fluorescence microscopy. Application of the hot needle test is not advised since it damages the particle and does not give information on chemical composition, although the application of the hot needle method is supported in cases where no other polymer identification method is available.

Particles are described by count, size classes, morphology, colours (optional) and polymer composition (on at least a subset). The minimum cut-off size for data to be reported is 300 µm (see section 4.3 for size classes).

3.6 Polymer identification

The determination of at least a subset of particles for their polymer composition via FTIR or Raman spectroscopy is mandatory.

Device settings and minimum library match (%) attributed is to be recorded within the metadata to EMODnet. Spectra libraries integrated for polymer composition determination should integrate spectra from synthetic and organic components. It is suggested to agree on one or several libraries that are used by all processing laboratories and/or to generate a combined FTIR and/or Raman spectra library for HELCOM microlitter monitoring.

It is recommended to analyse the polymer composition on a representative subset with a minimum of 10 % (preferably at least 20 particles) of synthetic particles identified within the size categories from 300 to 999 µm and from 1000 µm to 4999 µm. The subset size of particles identified in any smaller size category is to be discussed. The particles integrated in the subset are to be selected representatively according to size categories and morphologies.

4. Parameter and data recording

Parameters are to be recorded according to EMODnet requirements (see section 5). Data can also be reported to ICES DOME when parameters and attributes and e.g. harvesting of data from EMODnet will be harmonised (and the consent of the country is given). The reporting to or harvesting of data through ICES DOME is under discussion.

Parameters to be recorded encompass the following:

4.1 Numbers

The recording of the number of particles identified as synthetic polymers/microlitter is mandatory.

Data are calculated to the number of particles and optionally weighed in grams per volume of filtered water. At this stage, no recommendation on re-calculating number of particles into mass is given. The development of conversion algorithms based on polymer composition and particle size/volume is to be evaluated.

4.2 Morphology

The morphology of all identified particles is to be recorded according to the following morphology classes:

Table 1 Morphology classes to be used to report all identified particles.

EMODnet identifier "micro-litter morphology"	Name	Definition according to EMODnet	Definition according to GESAMP 2019 (Kershaw et al. 2019)
H0100004	Filaments	Slender thread-like micro-litter particles.	"Line" (Fibre, filament, strand): long fibrous material that has a length substantially longer than its width
H0100005	Films	Micro-litter particles derived from plastic sheets or thin plastic films.	"Film" (sheet): flat, flexible particle with smooth or angular edges
H0100006	Foams	Any kind of micro-litter particle made of plastic foam, including styrofoam.	"Foam" (EPS, PUR): near spherical or granular particle, which deforms readily under pressure and can be partly elastic, depending on weathering state
H0100002	Fragments	Irregularly-shaped plastic micro-litter particles with broken off edges that may be rounded or angular.	"Fragment" (granule, flake): irregular shaped hard particles having appearance of being broken down from a larger piece of litter
H0100003	Pellets	Micro-litter particles from industrial origin only. In comparison with granules, pellets are usually flat on one side, rough surface and irregular, round shapes.	"Pellet" (resin bead, Mermaids tears): hard particle with spherical, smooth or granular shape
H0100009	Granules	Micro-litter particles with smooth spherical shape. In comparison with pellets, they have a rounder shape	

It is under discussion whether microbeads are to be reported as a single class or identified from the data set as morphology: granules and the (smaller) dimension in size compared to pre-production resin pellets.

It is under discussion whether "pellets" and "granules" should be separate classes. In addition, it has to be considered that "film" and "foam" might not be identified due to restrictions of devices or protocols especially within smaller size fractions.

4.3 Particle dimensions

The dimensions of identified particles should be recorded according to the following size classes:

- 300 – 999 µm

- 1000 – 4999 µm

The reporting of size classes below 100 µm is optional according to the following size classes:

- 100 – 299 µm
- 50 – 99 µm
- 20 – 49 µm
- <20 µm

It is to be pointed out that results way be biased if particle dimensions are retrieved from mesh sizes from sieving and filtering or measuring actual length and width.

The reporting of absolute dimensions on particle length and/or particle width is optional. Sizes of particles are defined according to:

- Length (maximum Ferret diameter in longitudinal).
- Width (maximum Ferret diameter perpendicular to the identified length transect).

Fibres with a length >5000 µm are considered "mesolitter" and are therefore excluded from the data analysis.

4.4 Polymer composition

Polymer composition is to be reported according to polymer classes and is to be defined for at least a subset of identified synthetic particles.

It is suggested to align the polymer types according to the list provided and modified from AMAP 2021 (see Table 2) but to set up a short list with prioritised synthetic polymers that are predominantly found in environmental samples and that at least have to be reported when occurring.

Table 2: Polymer types for data reporting (modified from AMAP 2021).

Polymer type name	**Examples of materials included (detailed level)**	**Modifications compared to AMAP (2021)**
Acrylonitrile based	e.g. acrylonitrile butadiene styrene (ABS), polyacrylnitrile (PAN)	Modified to "Acrylonitrile based", PAN removed from polymer type and integrated here as an example
Cellulose based	e.g. cellulose acetate (CA), cellulose nitrate (CN)	Modified to "cellulose based", examples added
Polyamide based	e.g. all types of polyamide (PA) like various nylons	
Polycarbonate based	e.g. polycarbonate (PC)	Modified to "polycarbonate based"
Polychlorinated polymers	e.g. polyvinyl chloride (PVC), chlorinated PE, various chlorinated polymers	
Polyester based	e.g. polyethylene terephthalate (PET), all other types of polyesters	Modified to "polyester based"
Polyethylene based	e.g. high density polyethylene (HDPE), low density polyethylene (LDPE), and copolymers with a major PE fraction including ethylene-vinyl acetate copolymer (EVA)	EVA removed from polymer type and integrated into polyethylene based.
Polyfluorinated polymers	e.g. polytetrafluoroethylene (PTFE)	

Polymeth(ester)acrylate based	e.g. all types of polymeth(ester)acrylate (PM(ester)A)	
Polypropylene based	e.g. polypropylene (PP), and copolymers with a major PP fraction	
Polystyrene based	e.g. polystyrene (PS), and copolymers with a major PS fraction	
Polyurethane based	e.g. all types of polyurethane (PUR)	
Rubbers, automotive	e.g. styrene butadiene rubber (SBR), tire wear	SBR added as an example
Varnish/paint particles	If different from PM(ester)A	
Other plastics	e.g.polyether ether ketone (PEEK), polyoxymethylene (POM), polyvinyl acetate (PVA), polylactic acid (PLA), polyhydroxyalkanoate (PHA)	Examples added / moved from single polymer classes
Other rubbers	e.g. ethylene propylene diene monomer rubber (EPDM), nitrile rubbers, natural rubbers, silicone	Examples added / moved from single polymer classes / rubber types (refers to "rubbers sealing", "nitrile rubbers", "natural rubbers and derivates", "silicone rubbers and derivates"
Other microlitter materials	e.g. metal, glass	Examples added
Other semi-synthetic polymers	e.g. rayon	Polymer type added / introduced

4.5 Optional parameters

The recording of particle colours and/or transparency is optional. Colours and transparency are classified according to EMODnet:

Colour classes:

- black / grey
- blue / green
- brown / tan
- white / cream
- yellow
- orange / pink / red
- purple
- multicolour

It is suggested and discussed to include a class „colourless" in order to address particles derived from colourless and transparent foils or particles from e.g. (uncoloured) plastic bottles.

Transparency:

- Yes
- No

5. Data reporting

Data are to be reported to EMODnet according to current specification provided by EMODnet (i.e. Vinci et al. 2021).

The reporting to or harvesting of data through ICES DOME is under discussion.

The following lists comprise parameters (mandatory and optional), EMODnet codes and descriptions where available and suggestions for modifications or the integration of further parameters following the discussions and suggestions provided within these draft guidelines and first evaluations through EMODnet.

Parameters and related attributes are under continuous development. Therefore, it is recommended to consult the latest tables and vocabularies online at the NERC Vocabulary Server (NVS).

Table 3 Current list of default (green), mandatory (orange) and optional (light orange) parameters to be reported (modified from Vinci et al., 2021, p7)

Label/column header	Concept id	Use	Comments
Cruise		metadata/mandatory (ODV Default)	
Station		metadata/mandatory (ODV Default)	
Type		metadata/mandatory (ODV Default)	The suggestion is to use type "B". From manual: 'B' for bottle profile data. For time series and trajectories set to 'B' for small (<250) row groups
YYYY-MM-DDThh:mm:ss.sss		metadata/mandatory (ODV Default)	Start date/time. Format must be adapted to the date value (for example YYYY-MMDDThh:mm is second are not available)
Longitude [degrees_east]		metadata/mandatory (ODV Default)	start point coordinates
Latitude [degrees_north]		metadata/mandatory (ODV Default)	start point coordinates
LOCAL_CDI_ID		metadata/mandatory (ODV Default)	
EDMO_code		metadata/mandatory (ODV Default)	EDMO_CODE of the data centre distributing the data (the one connected to the CDI service)
MinimumObservation Depth [m]	MINWDIST	mandatory in ODV micro-litter	
MaximumObservation Depth [m]	MAXWDIST	mandatory in ODV micro-litter	
SampleID:INDEXED_TEXT	SAMPID01	mandatory in ODV micro-litter	
SamplingEffort [Km or L]	LENTRACK/ VOLWBSMP	mandatory in ODV micro-litter	The amount of effort expended during an event. It can be the survey distance from the beginning point in kilometres or a filtered volume in litres
Net_opening [cm]	MTHWDTH1	mandatory in ODV micro-litter	Net opening of the instruments used. This information is needed for the calculation of the covered surface in cm (e.g. diameter of the Ocean Pack RACE filtering “cakes” or bongo/manta net opening)
Mesh_size [micrometres]	MSHSIZE1	mandatory in ODV micro-litter	Mesh size of the filtering surface (e.g. manta or bongo net, filtering “cakes” of OceanPack RACE) in µm

Microlitter_Type:IND EXED_TEXT	SDN:H01	mandatory in ODV micro-litter	Type of the item (H01 SDN vocabulary); MLITYPS
Microlitter_Size:INDE XED_TEXT	SDN:H03	mandatory in ODV micro-litter	Size classes (H03 SDN vocabulary), MLITSZS
Microlitter_Count [Dimensionless]	MLITCNTW	mandatory in ODV micro-litter	Number of items collected. It's the official mandate from MSFD to provide the count of collected microplastics.
EventEndDateTime [YYYY-MMDDThh:mm:ss.sss]	ENDX8601	additional/optional	End date/time
EventEndLongitude [degrees_east]	ENDXXLON	additional/optional	End point coordinates. Either End Lat/Lon or SamplingEffort are mandatory
EventEndLatitude [degrees_north]	ENDXXLAT	additional/optional	End point coordinates. Either End Lat/Lon or distance are mandatory.
Microlitter length	NEW	additional/optional	
Microlitter width	NEW	additional/optional	
Microlitter_Weight [g]	MLDWWD01	additional/optional	Weight of the collected items, not mandatory Information in grams
Microlitter_Shape:INDEXED_TEXT	MLITSHPW	additional/optional	Shape of the item (H02 SDN vocabulary)
Microlitter_Color:IN DEXED_TEXT	MLITCOLW	additional/optional	Colour classes (H04 SDN vocabulary)
Microlitter_Transparency:INDEXED_TEXT	MLITROPW	additional/optional	Transparency classes (H06 SDN vocabulary)
Microlitter_Polymer_type:INDEXED_TEXT	MLITPOLW	additional/optional	Polymer type of the micro-litter (H05 SDN vocabulary)
WMO_Sea_State [Dimensionless]	WMOCSSXX	additional/optional	Sea conditions following the Douglas scale
Wind_direction [degT]	EWDAZZ01	additional/optional	Direction relative to true north from which the wind is blowing
Wind_speed [m/s]	WSBZZ01	additional/optional	Sustained speed of the wind (distance moved per unit time by a parcel of air) parallel to the ground at a given place and time.
Sampling_protocol	SAMPPROT	additional/optional	The name of, reference to, or description of the method or protocol used to produce the sample

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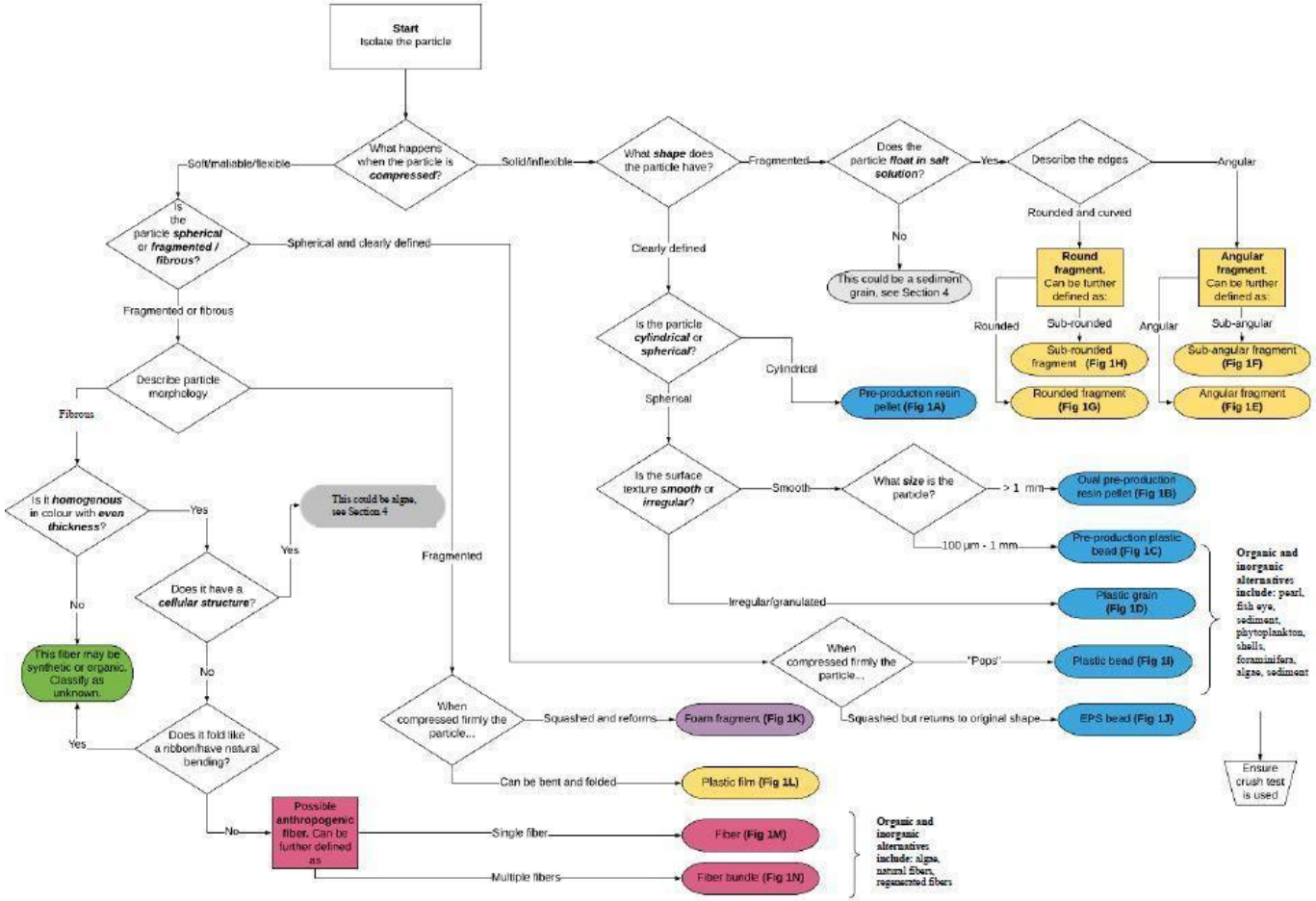
Annex

Figure A1 Proposed flow chart for the visual identification of microplastics. (AMAP, 2021, p223, reproduced from Lusher et al., 2020).



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A3.2 Annex 3

Specification of the prerequisites for the monitoring of microlitter in the water column and seabed sediments

For bibliographic purposes this document should be cited as: Specification of the prerequisites for the monitoring of microlitter in the water column and seabed sediments. HELCOM BLUES (2023)

2023

Specification of the prerequisites for the monitoring of microlitter in the water column and seabed sediments

The HELCOM guidelines for monitoring of microlitter in the water column and seabed sediments indicate the major prerequisites for future monitoring. These comprise:

Management of monitoring

- Designation of responsible institutions and laboratories for the planning and implementation of monitoring (sampling and laboratory analysis) and data transmission to EMODnet
- Provision of necessary resources

Sampling strategy

- Identification of monitoring stations including evaluation of possible association with existing other monitoring programmes
- Determination of monitoring frequencies and, if necessary, time of sampling (season)

Sampling

- Availability of ship and crew
- Sampling devices for water (i.e., Manta trawl) and seabed sediment (i.e., corer or grab sampler)
- Jars for samples and blank samples and further equipment (i.e., metal spoons, bowls, labelling material etc.)
- Sampling documentation

Sample treatment

- Assurance of laboratory measures to minimize contamination according to HELCOM guidelines
- Necessary laboratory equipment (i.e., glass beakers, watch glasses, sieves, lab balances, filtration unit, oven etc.)
- Consumables (i.e., filtered chemicals and water for sample digestion, density separation, sample transfer, filters)
- Reference material for reference samples

Sample analysis

- Availability of microscopes for particle identification and potential determination of particle dimensions and morphologies
- Availability of device for polymer identification (i.e., FTIR, Raman spectroscope etc.)

During the discussions within several technical workshops it became evident that several **hindrances and challenges for the monitoring of microlitter** in the water column and in seabed sediments still exist. These concern different topics and need to be solved within the future either providing further scientific evidence or by policy-makers and institutions being in charge for the monitoring. They are listed below:

- **different results depending on the laboratory conducting the analysis**:
    - these differences would be minimised if the same guidelines are applied;
    - also, if calibration studies are conducted among HELCOM countries where reference samples for the Baltic are prepared for the purpose. In order to fund them, applying for a COST action would be an option to consider, since participation in calibration studies, such as those organised by QUASIMEME is expensive;
- **cost of monitoring**:
    - there is a need of a ship for conducting the monitoring, including personnel (e.g., 3500€/day in Estonia);
    - manta trawling requires a lot of ship time which may incline countries towards sediment sampling instead;
    - it would be interesting to identify the possibility of sharing ships or laboratory use between countries;
    - further considerations are to be taken in relation to the number of sampling stations needed, including its cost implications;
- **further advances on research**:
    - other monitoring strategies may be considered in the long run, such as microplastics in rivers and/or river estuaries, close to potential sources;
    - once Baltic wide data are available from manta trawling, which is considered the current best option, there may be a need to consider conducting monitoring only in sediments, where results seem to be more comparable;
    - once more data are available, the number of stations needed to obtain a trend analysis may be smaller;
    - modelling is to be considered in conjunction with monitoring activities;
    - considerations are to be made on whether the results from ferry boxes flow systems are comparable to those from manta trawling.

The question of comparable data based on different libraries available (for example within FTIR or Raman analyses) to report polymers was also identified as a topic for further consideration. However, bearing in mind that the reporting is conducted by groups, this was not found to be an issue to prioritise.

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A3.2 Annex 4

Baltic Sea microlitter surface water data

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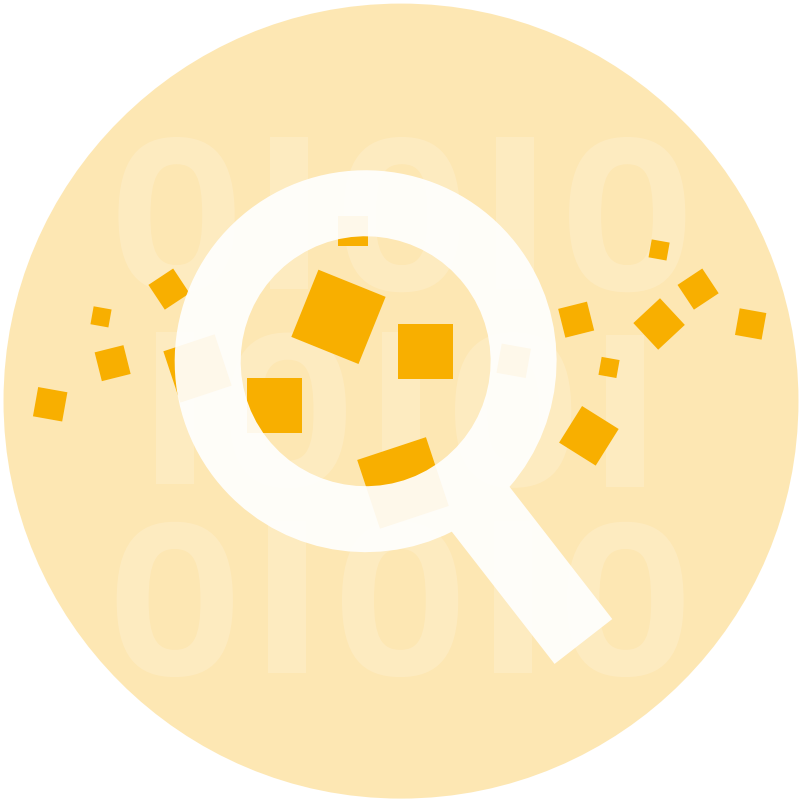

DOI	Area	Author	Publ. date	Sampling method	Mesh size, µm	Sampling speed, knots	Sampling time, min	Storing	Concentration	Unit	Visual analysis	Particle size	Polymers	Morphology	Colour	QA/QC
https://doi.org/10.3389/fmars.2022.875984	Eastern Baltic Sea	Mishra et al.	2022	Manta trawl	330	2	15-60 min	Formal-dehyde (37%)	0.11 - 0.65	items/m^3	Yes	Yes	Hot needle	fibers and fragments (films, foam, and pellets, fibrous plastics)	Yes	Yes/Yes
https://doi.org/10.1016/j.marpolbul.2021.112860	Gulf of Riga, Latvia	Aigars et al.	2021	Manta trawl	300	2-3	60	4-6 °C	0.09 - 4.43	items/m^3	Yes	Yes	ATR-FTIR	Fiber, fragment, film, bead, foam	Yes	Yes/Yes
https://doi.org/10.1016/j.envpol.2020.115700	Gulf of Finland, Finland (thin layers in the stratified)	Uurasjärvi et al.	2021	WP2 net	100	NM	nm	-20 °C	0 - 1.6	items/m^3	Yes	Yes	FTIR	Fiber, fragment	nm	Yes/Yes
									0 - 766	ng/m^3						
				Jussi sampler, 30 L	50				0.02 - 1.7	items/L						
									0 - 775	ng/L						
https://doi.org/10.1016/j.marpolbul.2021.112150	Baltic Sea, east of Bornholm, Open Baltic Sea,	Hänninen et al.	2021	Manta trawl	335	2.5	30	SDS	0.019 - 0.022	items/m^3	Yes	Yes	FTIR	Fiber, fragment, film	Yes	Yes/Yes
									3112 - 3554	items/km^2						
									0.003 - 0.004	items/m^2						
https://doi.org/10.1016/j.marpolbul.2020.111019	waters surrounding Sweden, Baltic	Schönlau et al.	2020	Manta trawl	333	1-3	60	4 °C	0 - 0.46	items/m^3	Yes	NM	NIR hyperspectral imaging	Fiber, fragment	Yes	nm/nm
				Pump, 20 m^3	300	nm	23-138		0 - 10.5							
					50				0 - 70.3							
https://doi.org/10.1007/s11356-019-07274-5	Gullmar fjord on the Swedish west coast	Karlsson et al.	2020	Manta trawl	300	nm	nm	nm	0.18 - 0.92	items/m^3	Yes	Yes	FTIR	fragments, expanded cellular plastics, air-filled spheres, fibers	Yes	nm/Yes
				Pump, 20 m^3				nm	0 - 0.4						nm	
https://doi.org/10.1016/j.scitotenv.2020.139493	Kiel Fjord, southwest Baltic Sea	Ory et al.	2020	Manta trawl	300	3	5	nm	0.0 -1.8	items/m^3	Yes	Yes	FTIR	Fragment	Yes	nm
https://doi.org/10.1016/j.marpolbul.2018.11.047	(in layers of stratified Baltic Sea)	Zobkov et al.	2019	PLEX bulk sampler	174	nm	nm	4-6 °C	15.4 - 79.1	items/m^3	Yes	Yes	µ-Raman	Fiber, fragment, film	nm	Yes/Yes
https://doi.org/10.1016/j.marpolbul.2017.10.049	Baltic Proper	Bagaev et al.	2018	Niskin bottles 0-217.5m, 10 and 30 L	174	nm	nm	room temp.	0.1 - 0.9	items/L	Yes	Yes	nm	Fibre, fragment, paint, non-plastic	Yes	Yes/
https://doi.org/10.1016/j.marpolbul.2018.01.066	South Funen Archipelago, Baltic Sea	Tamminga et al.	2018	Manta trawl	300	1.8-3.2	20	10 ml 37% HCl	0,04 - 0,09	items/m^3	Yes	No	Nile red	Fiber, fragment	nm	Yes/Yes
				Bulk sampler, 5L	5000, 1000, 300, <300	nm	nm	1 ml 37% HCl	1.03 ±0.80	items/L						
https://doi.org/10.1016/j.marpolbul.2017.04.062	Stockholm Archipelago, Baltic Sea	Gewert et al.	2017	Manta trawl	335	2-3	12-60	4 °C	1.56×10^4 - 6.18×10^5	items/km^2	Yes	Yes	FTIR	Fiber, fragment, paint flakes, macroplastics	Yes	Yes/Yes
									0.19 - 7.73	items/m^3						
https://doi.org/10.1016/j.marpolbul.2016.06.065	Gulf of Finland, northern Baltic Sea	Setälä et al.	2016	Manta trawl	333	2.5	10	nm	0.3 - 2.1	items/m^3	Yes	No	Hot needle	plastic fibres, plastic fragments, paint flakes, non-synthetic	nm	nm/No
				Submersable pump	300	nm	nm	nm	0 - 3.4							nm/Yes
					100	nm	nm	nm	0 - 8.2							

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A3.2 Annex 5

HELCOM BLUES case study: Microplastic concentrations in marine bottom sediments of the German Baltic Sea

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MRC
MICROPLASTIC RESEARCH AT CEN

HELCOM BLUES case study: Microplastic concentrations in marine bottom sediments of the German Baltic Sea

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Introduction

Activity 3 “Marine Litter” of the HELCOM BLUES project (HELCOM BLUES 2022) aims to promote the harmonisation of regional work on marine litter indicators and threshold values as well as ensuring alignment with the work of the EU’s MSFD Technical Group on Marine Litter (TG Litter). Therefore, guidelines on monitoring microlitter in seabed sediments and surface water have been drafted according to existing approaches and feasibility (HELCOM BLUES Microlitter Group 2022). These draft guidelines are currently applied as a case study to seabed sediments from the southern Baltic Sea.

Fig. 1: Sediment samples with digestion solution.

Study Area

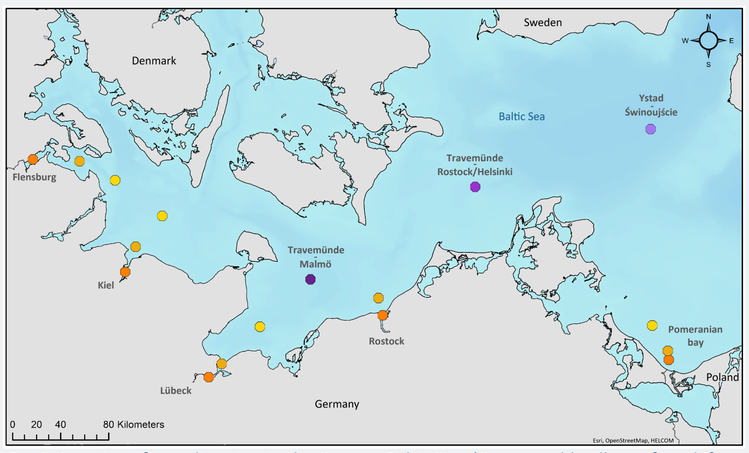

Fig. 2: Location of sampling sites in the German Baltic Sea. (Orange-gold-yellow = from left to right, stations with increasing distance to coastline. Purple = offshore stations).

Within the Baltic Sea region, a total of 29 grab sediment samples were taken in cooperation with Federal Agencies of Schleswig-Holstein and Mecklenburg Western-Pomerania in 2021 and 2022. Sampling sites (Fig. 2) were located in the region of Flensburg, Kiel, Lübeck, Rostock and Pomeranian bay. Three samples with increasing distance to the coastline were taken within each area. Three offshore stations within the Exclusive Economic Zone (EEZ) were also included in the study. Three parallel samples were taken in Rostock and the offshore stations to determine the variations of different grab samples.

Material & Methods

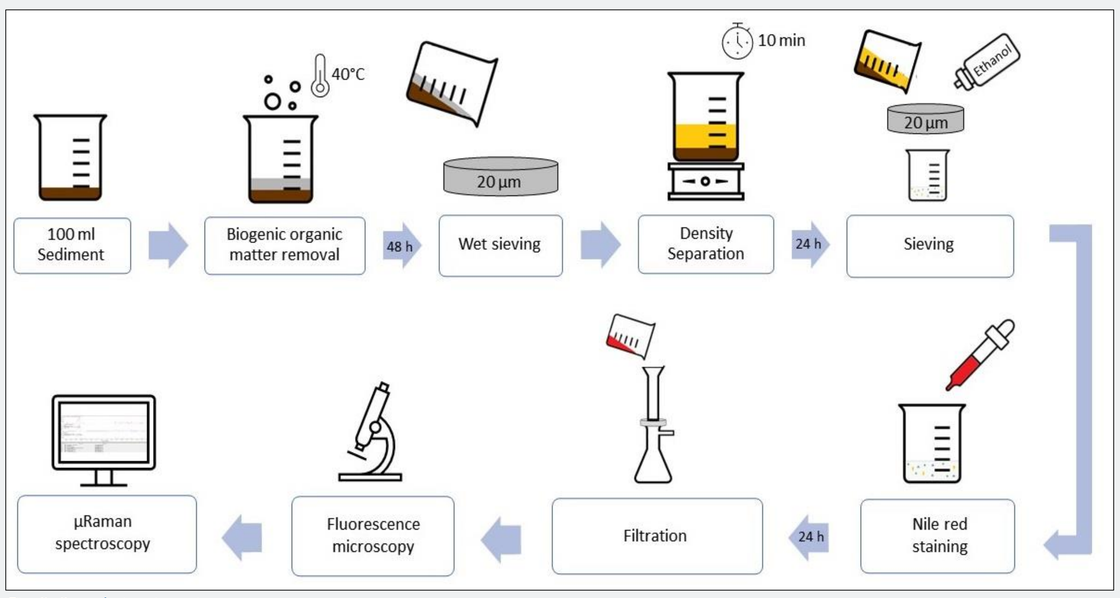

Fig. 3: Sample treatment.

Sample treatment

Removal of biogenic organic matter from the sediment was performed by adding a digestion solution (NaClO 6-14% and KOH 10M) (Fig. 1) that was allowed to stand at 40 °C for 48 hours with a subsequent wet sieving (20 µm). To extract the microplastic particles, a density separation was carried out. Therefore, NaI was added (density: 1.7 g/cm^3) at a ratio of 1:2, the sample was mixed with glass magnetic stirrers for 10 minutes and then left for settling for 24 hours. Then, 50 % of the supernatant was transferred over a 20 µm sieve and the sieving residue was captured within a beaker with ethanol. This was repeated twice to increase the efficiency. Sample suspensions were stained with Nile red (1 mg/ml in chloroform) and transferred to aluminium oxide filters (Anodisc 25, Whatman, 0.2 µm retention). Potential microplastic particles were detected via fluorescence microscopy (Axioscope 5/7 KMAT, Zeiss). A subset of particles is currently under investigation for polymer composition via µRaman spectroscopy (DXRxi2, Thermo Fisher Scientific) (Fig. 3). Along the microplastic analysis, sediment parameter such as water and organic matter content and grain size of the sediment is analyzed (in processing).

Recovery tests

To assess extraction efficiency, a reference sample was analyzed with each series of samples. For this purpose, a sand matrix was spiked with microplastic reference particles consisting of PET particles (125-200 µm). The recovery rate of a total number of 7 reference samples was > 60 %. Further processing to improve recovery is currently in progress.

QA/QC management

Precautions have been taken in order to reduce background contamination as much as possible. Therefore, glass and stainless-steel materials were used, all chemical solutions were filtered (691, VWR International, 1.6 µm retention) as well as the integration of field and procedural blanks. Between 3 and 8 particles per blank sample were found.

Results & Discussion

Microplastic concentrations and variations

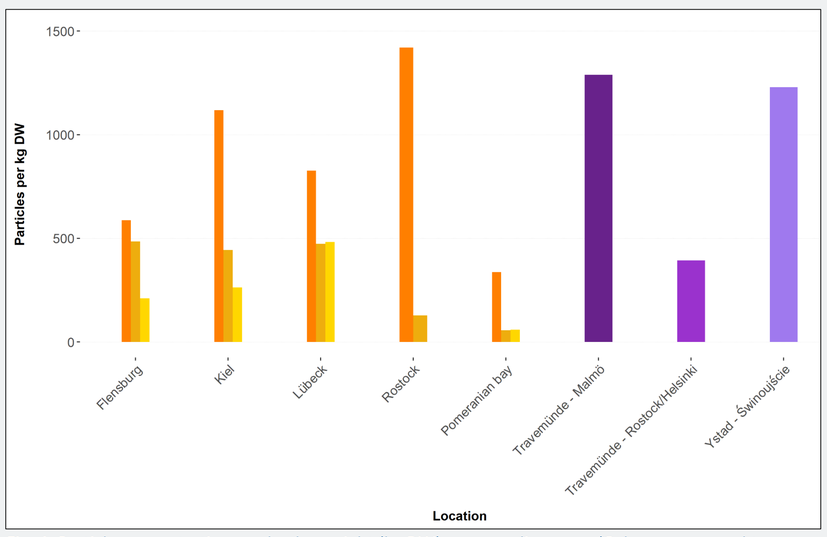

Fig. 4: Particle concentration per kg dry weight (kg DW) per sampling area. (Colors correspond to sampling points in Fig. 2).

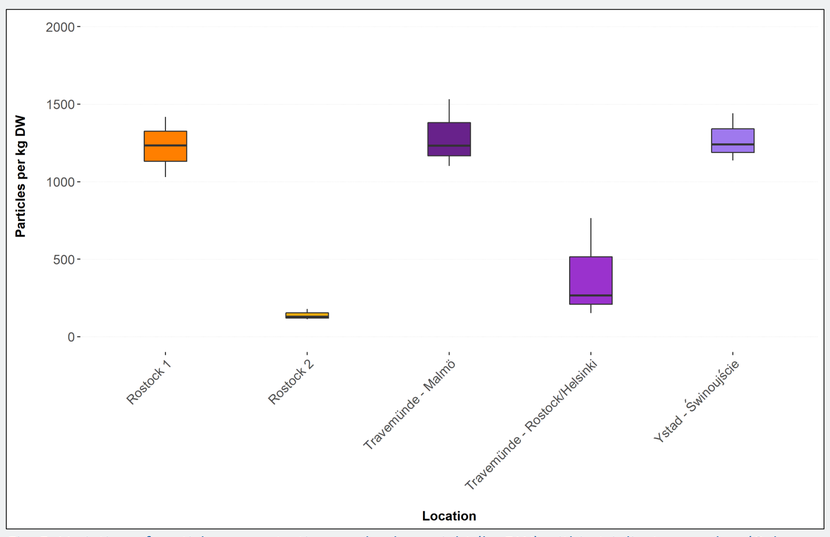

Fig. 5: Variation of particle concentration per kg dry weight (kg DW) within triplicate samples. (Colors correspond to sampling points in Fig. 2).

Particle size and morphology

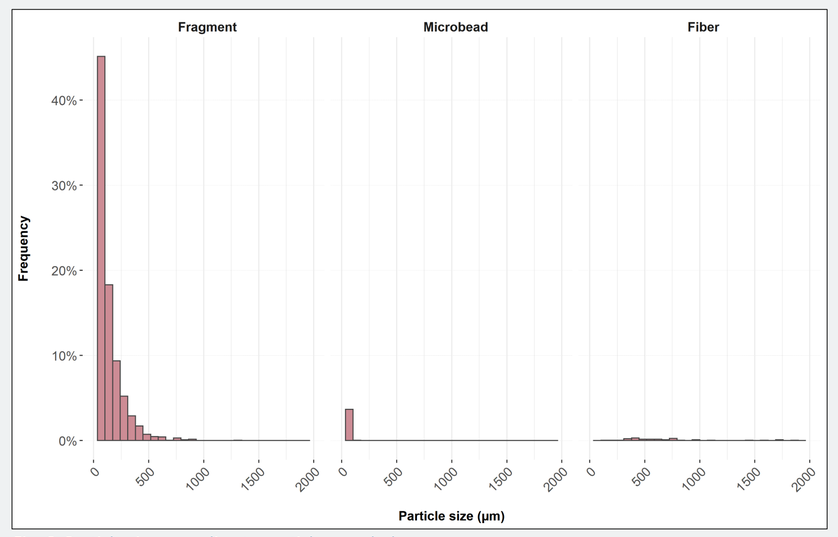

Fig. 6: Particle size according to particle morphology.

Figure 4 shows the microplastic particle concentration per kilogram dry weight (kg DW) of the different sampling sites. Within all samples, a total of 9,847 microplastics per kg DW are recorded. Regarding the coastal locations, the highest amount was found in the Rostock Warnow estuary with 1,420 microplastics per kg DW. Particle concentrations in the inner fjord and estuary area show the highest concentrations with decreasing particle numbers with increasing distance from the coastline for the stations Flensburg, Kiel and Rostock. In contrast, the two more remote sites of Lübeck bay and Pomerania bay show similar results. The higher amount of microplastic particles in fjords and estuaries has already been demonstrated in other studies (Harris 2020). For the offshore area, the Travemünde-Malmö site with 1,289 microplastics per kg DW shows the highest concentration. The sites are expected to be influenced by river inputs (Schmidt et al. 2017) or show distinct reduced flow velocities (Chubarenko et al. 2022). However, due to single sampling no significant differences can be calculated for the sampling sites and within the respective transect.

Particle concentrations of the triplicate sampling are shown in Figure 5. With 217 particles, the samples of the Travemünde-Rostock/Helsinki site have the highest variation. The Rostock samples have the lowest variation with 35 particles. Small-scale variations in sediment grain size and organic matter that are currently processed will be taken into account for further analysis of influencing factors.

Concerning the morphology of the particles, predominately fragments (92 %), followed by microbeads (6 %) and fibers (2 %) were found. The size frequency of the particles are shown in Figure 6. The fragments measure between 20 µm and 1316 µm (131 ± 121) with an increasing frequency at a decreasing particle size across all sites. The highest abundance of fragments are in the size class 20-100 µm.

Regarding microbeads, the measurements vary between 20 µm and 114 µm (43 ± 17). The highest concentration with 152 microbeads per kg DW were counted in the estuary area of Lübeck.

Conclusion

The sediment of the German Baltic Sea is contaminated with microplastics, with a tendency of decreasing microplastic concentration with increasing distance from the coast. Furthermore, higher concentrations are found again at the offshore stations. Triplicate samples show that different grab samples could result in different concentration amounts. Further analysis regarding the grain size and organic matter of the sediment need to be finalized to determine the influence on the particle concentration.

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