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Delegations will find attached document C(2024) 1459 final - Annex.

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ANNEX

ANNEX

to the

Commission Delegated Decision

supplementing Directive (EU) 2020/2184 of the European Parliament and of the Council by laying down a methodology to measure microplastics in water intended for human consumption

ANNEX

METHODOLOGY TO MEASURE MICROPLASTICS IN WATER INTENDED FOR HUMAN CONSUMPTION

1. Definitions

For the purposes of this Annex, the following definitions shall apply:

(1) 'microplastic' means a small discreet object that is solid, insoluble in water and is partially or wholly composed of synthetic polymers or chemically modified natural polymers;

(2) 'particle' means a minute piece of matter with defined physical boundaries;

(3) 'microplastic particle' means a microplastic object whose dimensions are equal to or less than 5 mm and whose length to width ratio is equal to or less than 3;

(4) 'microplastic fibre' means a microplastic object whose length is equal to or less than 15 mm and whose length to width ratio is greater than 3;

(5) 'polymer' means a substance consisting of molecules characterised by the sequence of one or more types of monomer units. Such molecules shall be distributed over a range of molecular weights wherein differences in the molecular weight are primarily attributable to differences in the number of monomer units. A polymer comprises the following:

   (i) a simple weight majority of molecules containing at least three monomer units which are covalently bound to at least one other monomer unit or other reactant;

   (ii) less than a simple weight majority of molecules of the same molecular weight.

(6) 'monomer unit' means the reacted form of a monomer in a polymer;

(7) 'synthetic polymer' means a polymer that is human-made material and which results from a polymerisation process which has not taken place in nature;

(8) 'microplastics concentration' means the quantity of microplastics present in water, expressed as the number of microplastic objects (particles and/or fibres) per cubic metre of water;

(9) 'natural polymer' means a polymer which results from a polymerisation process which has taken place in nature and is not chemically modified;

(10) 'microplastic particle size' means the area-equivalent diameter determined from an optical or chemical image of the microplastic;

(11) 'area-equivalent diameter' means the diameter of a circle having the same area as the 2-dimensional projection of the particle's optical or hyperspectral chemical images;

(12) 'microplastic fibre size' means the average value of the projected width of the microplastic fibre;

(13) 'insoluble polymer' means a polymer which has a solubility less than 2 g/L in water under thermal and chemical conditions relevant to water intended for human consumption;

(14) ‘priority polymers’ means the following polymers that are to be considered in the identification of microplastics:

(i) Polyethylene (PE);

(ii) Polypropylene (PP);

(iii) Polyethylene Terephthalate (PET);

(iv) Polystyrene (PS);

(v) Polyvinylchloride (PVC);

(vi) Polyamide (PA);

(vii) Polyurethane (PU);

(viii) Polymethylmethacrylate (PMMA);

(ix) Polytetrafluoroethylene (PTFE);

(x) Polycarbonate (PC);

(15) ‘polymer classification’ means analysed particles classified according to the following three categories:

(i) Identified as a priority polymer;

(ii) Identified as a synthetic polymer or a chemically modified natural polymer which is not in the list of priority polymers;

(iii) Other (e.g., minerals, natural polymer, other) or unidentified.

(16) ‘size classification’ means classification according to the area-equivalent diameter of microplastic particles in one of the following ranges:

(i) 20≤ area-equivalent diameter <50 μm;

(ii) 50≤ area-equivalent diameter <100 μm;

(iii) 100≤ area-equivalent diameter <300 μm;

(iv) 300≤ area-equivalent diameter <1000 μm;

(v) 1000≤ area-equivalent diameter <5000 μm.

(17) ‘filter cascade’ means a sequence of filters placed in series to collect particles from liquid flowing through the filters;

(18) ‘procedural blank’ means a sample that has been through the entire sampling, processing and measurement procedure and is analysed in the same manner as a normal sample but without having been exposed to the analyte;

(19) ‘vibrational spectroscopy’ means a technique used to measure the interaction of visible and infrared radiation with matter by absorption, scattering, or reflection;

(20) ‘Raman spectroscopy’ means a spectroscopic technique used to determine vibrational modes of molecules in solids, liquids and gases and based on illuminating a sample with a strong monochromatic light source and then measuring the portion of light which is in-elastically scattered from the material;

(21) ‘Infra-Red (IR) spectroscopy’ means a spectroscopic technique used to determine vibrational modes of molecules in solids, liquids and gases and based on measuring the interaction of infrared radiation with matter by absorption or reflection;

(22) ‘Fourier-Transform Infra-Red micro-spectroscopy (μ-FTIR)’ means a variation of infrared (IR) spectroscopy which combines a FTIR spectrometer with a microscope system for acquiring spatially resolved IR spectra and performing chemical imaging;

(23) ‘Raman micro-spectroscopy (μ-Raman)’ means a variation of Raman spectroscopy which combines a Raman spectrometer with a microscope system for acquiring spatially resolved spectra and performing chemical imaging;

(24) ‘Quantum Cascade Laser (QCL)-IR microscopy’ means a variation of Infra-red (IR) microscopy which utilizes a tuneable QCL as the IR source for acquiring spatially resolved IR spectra and performing chemical imaging.

2. Methodology to measure microplastics in water intended for human consumption

A filter cascade shall be used to collect particles and fibres from water intended for human consumption. Images from optical microscopy or chemical mapping are then used to determine individual particle size and shape, while vibrational micro-spectroscopy is used to identify particle compositions. The methodology shall be limited to particles with a dimension between 20 μm and 5 mm, and to fibres with length comprised between 20 μm and 15 mm. The methodology shall be used to determine the microplastics concentration expressed as the number of microplastics per cubic metre of water and concentrations of microplastics classified according to pre-determined size ranges, shape and composition categories.

(1) Samples shall be collected using filtration by passing water intended for human consumption through a cascade of four filters. The filters should be mounted in filter holders suitable for operating under positive pressure. The first filter, denominated (a), shall have a cut-off of 100 μm and the second filter, denominated (b), shall have a cut-off of 20 μm. The third filter, denominated (c), shall have a cut-off of 100 μm and the fourth filter denominated (d) shall have a cut-off of 20 μm. Filters (a) and (b) shall serve to collect the suspended matter from the water intended for human consumption. Filters (c) and (d) shall be used, where required, to produce procedural blanks to assess levels of microplastic contamination, in particular from laboratory equipment, reagents and surrounding atmosphere, occurring during the steps of sampling, treatment and analysis. To minimise atmospheric contamination of samples, the required volume of water should be piped directly from the sampling point through the filter cascade without the use of an intermediate collection or storage vessel. Intermediate collection/storage vessels may only be used when immediate, direct cascade filtration at the sampling point is impossible or impracticable, notably for technical or safety reasons.

(2) During all steps of collecting, treating, storing and analysing samples, all reasonable precautions shall be taken to avoid contamination of the samples with extraneous plastic particles from the surrounding environment, personal protective or laboratory equipment. All liquids used in sample processing shall be filtered (0.45 μm or less) prior to use.

(3) A minimum volume of 1 000 (thousand) litres of water shall be sampled. The total volume of water passed through the filter cascade shall be measured and recorded.

(4) A sample analysis by vibrational micro-spectroscopy may be done directly on the original collection filters, if they are compatible with the analytical method used. Incompatibility of the original collection filter may be due to insufficient smoothness of the filter surface, interference from scattered signals from the filter, fluorescence or absorption of optical signals when used in transmission.

(5) If a sample analysis cannot be done directly on the collection filter, the particulate materials may be re-suspended in liquid and transferred to an alternative support for subsequent analyses. If necessary, density separation and/or chemical/enzymatic treatment measures may be applied to reduce the presence of non-plastic materials such as minerals, metal oxides and natural organic matter.

(6) Experimental verifications shall be performed to assess the recovery of material on each of filters (a) and (b) when applying the methodology as implemented by the user. This may be done by spiking the water flow into the filter cascade sample with a known quantity of clearly identifiable microplastics and verifying the quantity recovered following the analysis procedure. The spikes shall include particulates with sizes, densities and numbers appropriate for assessing recovery on filters (a) and (b). It is recommended to use spike particles in the size range from 120 to 200 μm to assess the recovery on filter (a). To assess recovery on filter (b) it is recommended to use particles in the size range from 30 μm to 70 μm. Recovery shall be assessed using particles of at least two of the priority polymers. The polymers used shall include at least one with higher density than water (e.g., PET) and at least one with lower density than water (e.g., PE). In each case, the number of spike particles shall be within the range of 50 to 150. The analysis procedure shall be considered acceptable if the recovery rate is within the range of 100% to +/- 40%.

(7) When material is transferred from collection filters (a) or (b) to an alternative analytical support (secondary filter or other appropriate surface) this shall preferably be done without sub-sampling. If the analytical procedure includes sub-sampling steps, then the final analysed sample shall represent at least 10% of the material recovered from the original volume of water sampled. Analysis shall be done separately on materials collected on each of the filters (a) and (b).

(8) Filters (c) and (d) shall be used to produce procedural blanks. The procedural blank produced with filter (c) shall consist of a 100 μm filter and shall be subjected to the same processing and analysis steps as collection filter (a). The procedural blank produced with filter (d) shall consist of a 20 μm filter and shall be subjected to the same processing and analysis steps as collection filter (b). To quantify the typical levels of background contamination occurring during the performance of the analytical procedures, it is recommended to collect, process and analyse a minimum of ten procedural blanks of each filter type. These values shall be used to calculate the mean (μ) and standard deviation (σ) of the background microplastic contamination. Subsequently, further procedural blanks shall be collected periodically and analysed to monitor variations in the level of background contamination. If any periodic blank exceeds the mean background contamination (μ) by more than three times the standard deviation (σ) then the laboratory shall investigate the source of the increased contamination and take measures to reduce it.

(9) Prior to undertaking analyses by vibrational spectroscopy, optical microscopy or chemical mapping shall be used to measure or estimate the number of generic particles (≥20 μm) on the full filter or sample support. Where the total number of generic particles on the filter is too high to measure in a practical time, the operator may limit the analysis to one or more smaller sub-areas of the filter: the selection of the area shall follow appropriate sub-sampling strategies which maintain a representative sample. The sub-sampling shall cover at least 20% of the area of the sample support or filter. Where sub-areas of the filter are used, the operator shall analyse all particles and fibres in the size range ≥20 μm.

(10) The compositional analysis of microplastic particles and fibres shall be carried out using vibrational spectroscopy methods such as μ-FTIR, μ-Raman or equivalent variations such as QCL-IR. The instruments shall be capable of acquiring IR/Raman spectra from

particles within the size range of 20 μm or less. Optical images or chemical maps shall be used to determine the size of microplastic particles and fibres. Optical images shall be acquired using an objective of at least 4x magnification. The particle size classification shall be based on area-equivalent diameter whenever this option is available to the instrument operator. Alternative measures of diameter shall be used only if this option is not available. The type of alternative diameter shall be reported.

(11) The identification of particles and fibres from acquired spectra shall be carried out by comparison with spectra of known materials contained in a spectral library. The spectral library used for identification shall contain examples of all the priority polymers and shall in addition contain examples of proteins and minerals and natural polymers such as cellulose that might commonly be present in water intended for human consumption.

(12) Where automated identification procedures are used, an experimental verification shall be performed to assess the appropriate positive acceptance criteria for spectrum matching. The verification shall consider the specific features of the applied instrumentation, spectral library and identification strategy. This may be done by using pure polymer microparticles, but the evaluation has to cover the relevant size ranges to be retained by the sampling filters, notably, (a) >100 μm and (b) 20-100 μm. Once the minimum quality level applied for positive spectral identification has been established, that level shall remain fixed for the protocol applied by the analytical laboratory.

(13) Data shall be recorded separately from the materials collected on each of the two collection filters (100 μm and 20 μm cut-off). Where procedural blank samples are collected data shall be recorded separately from the materials collected on each of the blank filters (20 μm or 100 μm cut-off).

(14) Measurement requirements: the filter or sub-area of the filter shall be analysed so as to examine all microplastic particles and fibres as defined in the size ranges detailed in section 1, points (3) and (4).

(15) Data acquired on microplastic particles and fibres shall be elaborated to categorise each object on the basis of its size, number, shape and composition as follows:

(a) shape: particle or fibre according to the definitions in section 1, points (3) and (4)

(b) size (if particle): the size category listed in section 1, point (16);

(c) composition (if particle): identified as a priority polymer as defined under Section 1, point (14) or identified as a non-priority polymer under section 1, point (15)(ii) or identified as other material under section 1, point (15)(iii);

(d) polymer type (if fibre): where fibre dimensions and instrument capabilities allow for a positive identification of polymer type, this shall be identified in accordance with the categories defined in section 1, points (14) and (15) otherwise it shall be indicated as unidentified fibre.

(16) If the analysis of the materials on the filters or sample support does not address all collected particulates (e.g., due to sub-sampling) in the relevant size range, the data shall be appropriately scaled so as to correctly represent the concentration of microplastics in the original sample of water intended for human consumption. The content of microplastics in water intended for human consumption shall be expressed as the number of microplastic particles or fibres per cubic metre.

(17) Users of this methodology shall ensure that all of the following additional information is recorded in relation to each sample collected and measured:

(a) total volume of water sampled;

(b) location and time of sampling and sample analysis;

(c) sample treatment details;

(d) spectroscopic method and instrument applied;

(e) details of any sub-sampling during analysis or sample preparation;

(f) chemical nature of any plastic component(s) in sampling device or in equipment used during sample preparation;

(g) any deviation from the methodology including justification.

(18) When using this methodology, standard laboratory and environmental safety rules shall apply.