



# Microplastics: Methods and analysis packages

ALS has been offering analysis of microplastics in water since 2007. We are now also able to offer analysis of microplastics in more complex sample types such as soil, sludge, sediment, biota (e.g. fish and mussels) and food (e.g. sea salt, powdered milk, tea). The separation technique to extract the particles varies depending on the sample type. Generally, inorganic material is removed after flotation, degradation of organic material is performed by enzymatic treatment and oxidation before filtration. The analysis is performed by  $\mu$ FTIR (Fourier Transform Infrared Spectroscopy).

## Microplastics in pure water

When analyzing microplastics in pure water such as drinking water, the sample is filtered through a 20  $\mu$ m pore metal mesh. Analysis of the filter is performed with  $\mu$ FTIR. Particles with diameters between 5 mm and 20  $\mu$ m are counted and identified with the exception of black particles. Black particles are counted and identified between 5 mm and 40  $\mu$ m.

Analysis packages:

*A-7a-Plus Microplastics inclusive black particles in pure water*

*A-7a-Bas Microplastics exclusive black particles in pure water*

## Microplastics in contaminated water

When analyzing microplastics in dirty water such as wastewater, surface water and storm water, hydrogen peroxide is added to the sample and allowed to stand for 24 hours under stirring and low heat. Mineral particles are removed from the sample by density separation in a saline solution. The solution is filtered through metal mesh with pore size 500  $\mu$ m.

Materials that do not pass the filter are examined ocularly and any microplastics are collected for further analysis with  $\mu$ FTIR. Fenton's reagent is added and the solution is allowed to stand for the next day under stirring and temperature control. The sample is filtered through a 40  $\mu$ m metal mesh pore.

Analysis of the filter is performed with  $\mu$ FTIR. Particles with diameters between 5 mm and 40  $\mu$ m are counted and identified with the exception of black particles. Black particles are counted and identified between 5 mm and 40  $\mu$ m.

Analysis packages:

*A-7b-Plus Microplastics inclusive black particles in wastewater*

*A-7b-Bas Microplastics exclusive black particles in wastewater*

## Microplastics in soil & sediment

When analyzing microplastics in soil and sediment, a representative subsample is taken for dry matter determination. The remaining portion of the sample material is dried at 50°C. Dry material is screened through metal mesh with 5 mm mesh width and then

metal mesh with 1 mm mesh width. Material that does not pass 5 mm is removed.

Materials that do not pass 1 mm screen are examined ocularly and any microplastics are collected for further analysis with FTIR. A representative sub-sample is taken from the screened material. Hydrogen peroxide is added to the sample and allowed to stand for a day under stirring and low heat. Mineral particles are removed from the sample by density separation in a saline solution. The solution is filtered through metal mesh with pore size 500  $\mu$ m. Materials that do not pass the filter are examined ocularly and any microplastics are collected for further analysis with  $\mu$ FTIR. Fenton's reagent is added and the solution is allowed to stand until the next day under stirring and temperature control. The sample is filtered through a 40  $\mu$ m metal mesh pore.

Analysis of the filter is performed with  $\mu$ FTIR. Particles with diameters between 5 mm and 40  $\mu$ m are counted and identified with the exception of black particles. Black particles are counted and identified between 5 mm and 40  $\mu$ m.

Analysis packages:

*A-7c-Plus Microplastics inclusive black particles in soil, sediment*

*A-7c-Bas Microplastics exclusive black particles in soil, sediment*

## Microplastics in sludge

When analyzing microplastics in sludge, a representative sub-sample is taken for dry matter determination. The remaining portion of the sample material is dried at 50 °C. Dry material is screened through metal mesh with 5 mm mesh width and then metal mesh with 1 mm mesh width. Material that does not pass 5 mm is removed.

Materials that do not pass 1 mm sieve are examined ocularly and any microplastics are collected for further analysis with FTIR. A representative sub-sample is taken from the screened material. Hydrogen peroxide is added to the sample and allowed to stand for a day under stirring and low heat. To the sample solution is added pH-regulating buffer and enzyme to catalyze the degradation. The solution is allowed to stand for 24 hours under stirring and low heat (max 50 °C). Mineral particles are removed from the sample by density separation in a saline solution. The solution is filtered



through metal mesh with pore size 500 µm. Materials that do not pass the filter are examined ocularly and any microplastics are collected for further analysis with µFTIR. Fenton's reagent is added and the solution is allowed to stand for the next day under stirring and temperature control. The sample is filtered through a 40 µm metal mesh pore.

Analysis of the filter is performed with µFTIR. Particles with diameters between 5 mm and 40 µm are counted and identified with the exception of black particles. Black particles are counted and identified between 5 mm and 40 µm.

Analysis packages:  
A-7d-Plus Microplastics inclusive black particles in sludge  
A-7d-Bas Microplastics exclusive black particles in sludge

### Microplastics in biota

When analyzing bioplastics in biota such as fish and mussels, the sample material is dissected, dispersed and homogenized. Hydrogen peroxide is added to the sample and allowed to stand for a day under stirring and low heat. To the sample solution is added pH-regulating buffer and enzyme to catalyze the degradation. The solution is allowed to stand for 24 hours under stirring and low heat (max 50 °C). Mineral particles are removed from the sample by density separation in a saline solution. Fenton's reagent is added and the solution is allowed to stand for the next day under stirring and temperature control. The sample is filtered through a 40 µm metal mesh pore.

Analysis of the filter is performed with µFTIR. Particles with diameters

between 5 mm and 40 µm are counted and identified with the exception of black particles. Black particles are counted and identified between 5 mm and 40 µm.

Analysis packages:  
A-7f-Plus Microplastics inclusive black particles in biota  
A-7f-Bas Microplastics exclusive black particles in biota

### Microplastics in food

When analyzing microplastics in food as for example sea salt, powdered milk, honey, tea and beer organic material is degraded by enzymes similar to our natural digestive enzymes. For oxidation, hydrogen peroxide and Fenton's reagent is added before filtration. The sample is filtered through a 40 µm metal mesh pore.

Analysis of the filter is performed with µFTIR. Particles with diameters between 5 mm and 40 µm are counted and identified with the exception of black particles. Black particles are counted and identified between 5 mm and 40 µm.

Analysis packages:  
A-7f-Plus Microplastics inclusive black particles in food  
A-7f-Bas Microplastics exclusive black particles in food

Matrix	Drinking water	Wastewater	Soil, sediment	Sludge	Biota	Food
Package	A-7a-Plus/-Bas	A-7b-Plus/-Bas	A-7c-Plus/-Bas	A-7d-Plus/-Bas	A-7e-Plus/-Bas	A-7f-Plus/-Bas
Amount	1 liter	500 ml	500 g	500 g	100 g	100 g
Accreditation	no	no	no	no	no	no
Report limit	5 particles/ liter	25 particles/ liter	25 particles/ 100 g	25 particles/ 100 g	25 particles/ 100 g	25 particles/ 100 g
Analysis FTIR	yes	yes	yes	yes	yes	yes
Black particles	yes	yes	yes	yes	yes	yes
Ox, H <sub>2</sub> O <sub>2</sub>	--	yes	yes	yes	yes	yes
Ox, Fenton	--	yes	yes	yes	yes	yes
Enzyme	--	--	--	yes	yes	yes
Separation	--	yes	yes	yes	yes	no
Filter 20 µm	yes	--	--	--	--	--
Filter 40 µm	--	yes	yes	yes	yes	yes
Filter 500 µm	--	yes	yes	yes	--	--
Sieving 1 mm	--	--			--	--
Sieving 5 mm	--	--			--	--
Dry weight	--	--	yes	yes	--	--
Drying	--	--			--	--
Sample prep. biota	--	--	--	--	yes	yes

Table 1: Summary of our packages for analysis of microplastics. Table explanation is found on next side.

## Table explanation:

**Matrix:** ALS offers analysis of microplastics in drinking water, wastewater, soil, sediment, sludge, fish and clams.

**Packages:** ALS offers five packages for analysis of microplastics. The packages are compiled to cover interesting sample types. The price for the different packages varies as the time required for sample preparation varies for different sample types.

**Amount:** The amount of sample material from the customer should be about 2-5 times more than what is used in the analysis. The remaining sample material is stored for 4 weeks for water and 8 weeks for solid material. For any re-analysis or for other analysis.

**Accreditation:** The methods for the analysis we offer for microplastics are not accredited, however, the analysis are included in our quality assurance system.

**Analysis:** Polymer type identification and quantification is performed with  $\mu$ FTIR (Fourier Transform Infrared Spectroscopy). Particles with diameters between 5 mm and 40  $\mu$ m (20  $\mu$ m for drinking water) can be counted and identified with the exception of black particles. Black particles are counted and identified between 5 mm and 80  $\mu$ m.

**Black Particles:** Black particles (eg tires) require the use of ATR (Attenuated Total Reflectance) technology in the analysis. When analysing with ATR technology, each particle must be analysed manually, one at a time, which takes extra time. Black particles are counted and identified between 5 mm and 80  $\mu$ m. A price increase may be applicable for samples containing black particles.

**Reporting limit:** The reporting limit for pure water is 5 particles per liter and for other sample types 25 particles per liter or per 100 gram for materials. The detection limit can be raised if the matrix is difficult e.g. if the sample contains organic material that is difficult to break down.

**Oxidation with H<sub>2</sub>O<sub>2</sub>:** Hydrogen peroxide is added to the sample solution and allowed to stand for one day under stirring and low heat (max 50 °C).

**Oxidation with Fenton's reagent:** Hydrogen peroxide is added to the sample solution, pH is adjusted with sodium hydroxide and Iron (II) is added as a catalyst. The solution is allowed to stand until the next day under stirring and temperature control (max. 25°C).

**Enzymatic treatment:** Enzymatic treatment includes treatment with cellulase and / or protease. To the sample solution is added pH-regulating buffer and enzyme to catalyze the degradation. The solution is allowed to stand for 24 hours under stirring and low heat (max 50 °C).

**Separation:** Mineral particles are separated from the sample by flotation in zinc chloride solution with the density of 1.5 kg/L. After separation, the mineral-containing fraction is separated and the residue is collected for further processing. The separation solution is recycled for the next separation.

**Filters:** Filters used in sample preparation and analysis consist of stainless steel metal mesh with pore sizes 20, 40, 500  $\mu$ m. Filters used for analysis with FTIR are 20  $\mu$ m for drinking water and 40  $\mu$ m for other sample types.

**Drying / sieving / TS:** A representative sub-sample is taken for dry matter determination. The remaining portion of the sample material is dried at 50 °C. Dry material is screened through metal mesh with 5 mm mesh width and then metal mesh with 1 mm mesh width. Material that does not pass 5 mm is removed. Materials that do not pass 1 mm sieve are examined ocularly and any microplastics are collected for further analysis with FTIR. A representative sub-sample of 50 g is taken from the screened material for further processing.

**Sample preparation biota:** Sample preparation of fish and mussels includes dissection, dispersion and homogenization.



You are always welcome to contact our client service for more information about our analyses of microplastics!



ALS Scandinavia AB  
Rinkebyvägen 19c  
182 36 Danderyd

phone: 08-527 752 00  
e-post: info.ta@alsglobal.com

[www.alsglobal.se](http://www.alsglobal.se)

version 9-11-2020