Patented microliter filter crucible enables time-efficient and cost-effective microplastic analytics

Filtration and detection in a single element

Scientists across the globe are investigating the effects of microplastics finding their way into the environment. One key aspect of this work is assessing the potential risks for humans and the environment. However, the lack of uniform, quick, and practicable methods for taking samples and performing analyses makes it difficult to compare the results currently available. This situation is compounded further by the fact that the World Health Organization (WHO) only rates very few studies as truly reliable in its latest report, *Microplastics in drinking water*. With the German Federal Institute for Materials Research and Testing (BAM), the German Federal Environmental Agency (UBA) and the leading international technical weavers GKD – Gebr. Kufferath AG (GKD), a powerful trio from the fields of research, regulatory authorities, and industry have now taken a key step toward establishing a fast and secure standard procedure for microplastic analytics. Their joint development – a patented microliter filter crucible with Optimized Dutch Weave (ODW) from GKD for determining the mass content of microplastics in environmental media – facilitates fast and reliable routine analyses, even with complex samples.

The BAM, UBA, and GKD have been engaged in collaborative research for more than three years on various projects, sponsored by the German Federal Ministry of Education and Research (BMBF), to determine the levels of microplastics in various bodies of water, as well as developing sampling strategies and methods of analysis. Based on the issue at hand, the results

required to address it, and the type of environmental medium to be sampled, experts from the fields of science and practical implementation (economy or authorities) employ two different approaches: spectroscopic or thermoanalytical methods. The widely used spectroscopic approach, for example µ-Raman or µ-infrared spectroscopy, determines not only the particle size, but also the number, shape, and type of particles present. However, one major disadvantage of these approaches is that preparing real samples is both a time-consuming and labor-intensive process. This creates an obstacle for the kind of high sample throughput required for routine procedures – such as monitoring. The situation is made more acute by the fact that, for technical reasons, the spectroscopic analyses employed are limited to particles smaller than ten micrometers. This limits the usefulness of the results gained from the analyses – for example when investigating mineral water for microplastic particles with potential relevance at the human-toxicological level. The German Federal Institute for Materials Research and Testing (BAM) and the German Federal Environmental Agency (UBA) are therefore committed to using thermoanalytical procedures, in particular the TED-GC/MS (**T**hermal **E**xtraction **D**esorption **G**as **C**hromatography **M**ass **S**pectrometry) method of analysis, which was developed specifically for this purpose. In contrast to spectroscopic methods, TED-GC/MS determines mass content in solid environmental samples both quickly and reliably – and generally without the need to prepare the sample first. It should be possible to capture particles down to one micrometer in the near future using the microliter filter crucible developed for this method together with GKD. This would open up a whole range of options in the regulatory field. European legislative initiatives suggest that the first step in future provisions will focus on total content in milligrams per liter or per kilogram.

Stainless steel filter crucibles with selectivity < 10 µm for complex samples

The patented TED-GC/MS solution comprises three components that intelligently combine two familiar and fast analytical techniques. These are thermogravimetric analysis (TGA), as a standard analytical method for polymers, and gas chromatography/mass spectrometer (GC/MS) analysis, which is commonly used in trace analyses. These two components are linked via a solid phase extraction process, thereby guaranteeing a high throughput of complex environmental samples. Under inert conditions, the samples are decomposed and the specific decomposition products of the microplastic particles are collected on the solid phase absorber. This is then transferred to the GC/MS in a fully automated process. Here, these specific decomposition products are desorbed, separated by gas chromatography, and recorded in the mass spectrometer. Based on their characteristic chemical structure, they can be quantified and categorized as various polymers and ultimately microplastic types. However, the path to achieving this is rather laborious. First of all, samples need to be taken that contain a representative proportion of microplastic particles. In the case of water or air samples, this is generally achieved through filtration of the media. Microplastics from soil samples or sediments also need to be concentrated in a density separation step and the supernatants need to be filtered out of the solution. The samples must then be dried and transferred into the sample crucible used for TED-GC/MS analytics. All of these steps involve the risk of particle loss and contamination. They must also be performed using plastic-free equipment in plastic-free ambient conditions.

This is where the German Federal Institute for Materials Research and Testing (BAM), the German Federal Environmental Agency (UBA), and GKD come in. Their idea is to replace the aluminum oxide microliter filter crucible present in the system with an innovative crucible that also serves as a filter for environmental samples. The new filter crucible can be used directly for

filtration of the sample media or flotation items, thereby eliminating the need for additional work steps and minimizing the risks of particle loss and contamination. The Optimized Dutch Weave developed by GKD within the scope of the *OEMP project* (**O**ptimized Materials and Process for **E**limination of **M**icro**p**lastics from the water cycle), sponsored by the BMBF, with a geometric pore size of six micrometers (ODW 6) represented the basis for this. This stainless steel fabric is characterized by a single-ply design that is produced from a particularly high number of superfine warp and weft wires. The apertures in the slot-shaped pore geometry on the smooth mesh surface are smaller than inside the fabric. This type of fabric therefore guarantees an exceptionally high particle retention and flow rate. Unlike the plastic fabric still used in the majority of wastewater management applications, the rugged stainless steel construction is also suitable for high volumetric flows and does not cause any contamination due to plastic abrasion. At the same time, the ODW 6 fabrics guarantee determination of the bubble point via CFD thanks to a process developed at GKD. This means that all particles above the separation limit of six micrometers are reliably filtered out. In its role as project manager in the OEMP project, GKD collaborated with both Dr. Ulrike Braun, Team Lead in the Physical & Chemical Analysis of Polymers Department at the Federal Institute for Materials Research and Testing (BAM), and Dr. Claus Gerhard Bannick, Head of Wastewater Technology Research at the German Federal Environmental Agency (UBA), for the first time. In this context, both researchers were also able to familiarize themselves with and came to appreciate the ODW 6 developed by GKD.

The microliter filter crucible developed jointly within the scope of the *RUSEKU* follow-up project, which focuses on representative investigation strategies for an integrative system understanding of specific entry of plastics into the environment and is sponsored by the BMBF, has a diameter of eight millimeters and a height of ten millimeters. Its base is made from Optimized

Dutch Weave with a geometric pore size of five micrometers (ODW 5) and is welded onto the fixture body. The crucible is still in the prototype stage, but GKD has already defined the next steps for large-scale series production. For example, the necessary leak-tight integrity of the design from GKD is to be confirmed in bubble point testing of every single crucible by bombardment with liquid. An individual QR code that is lasered onto the crucible contains a link to a website, where this test report will be made available to users. This crucible-specific labeling also eliminates any risk of confusion or mix-ups when a large number of samples are being analyzed. For Ulrike Braun, this proactively developed solution once again exemplifies the high degree of identification of the technical weavers with their product, as well as the trust in the expertise of both her and Claus Gerhard Bannick. A trusting and constructive cooperation has developed among the partners over the years of project collaboration. The scientist really appreciates "the willingness displayed by GKD to try out new things without getting caught up in lots of worries". Claus Gerhard Bannick is also keen to express his praise: "GKD has an excellent ability to innovate and is also willing to take risks in this regard." The filter function of the microliter filter crucible also means that the high-risk process of sample transfer and the time-consuming work steps of freeze-drying are no longer required. With selectivity of five micrometers, the microliter filter crucible performs the final filtration step and is then used, together with the filter cake, in the TGA of TED-GC/MS in an automatic process.

Tried and tested in one year of practical operation

In the year since it has been in use, both Ulrike Braun and Claus Gerhard Bannick have already been able to gain valuable practical experience with the microliter filter crucible in the field of microplastics analytics with their teams. The team from the German Federal Institute for Materials Research and Testing (BAM) first tested the crucible with mineral water from PET

bottles for the patent office during the patent application. "The matrix of bottled water is very simple. The analysis worked really well with the crucible as the sampling container," remembers Ulrike Braun, adding that "the required results were ready within just a few hours." Claus Gerhard Bannick confirms that his experience has also been positive, as the results of six samples and two blind samples for various water-based beverages – water, cola, lemonade – in a quality control process were ready after just one day. "We would have needed significantly longer time frames to perform this spectroscopically," adds Bannick. This explains his belief that the microliter filter crucible could be the key to delivering a long-awaited time-efficient and cost-effective solution for routine analyses. "Anything that takes longer than 120 hours from sampling to the final results simply has no chance in practice," adds the expert. He therefore sees broad application potential for the crucible with various benefits based on the specific testing task. Testing of treated wastewater or surface water in the quality control of various purification processes are prime examples of this for him. From a purely technical perspective, Bannick sees another deployment area for the crucible following density separation, for example in analysis of organic fertilizers, soils, or sediments. Should more extensive requirements be implemented in directives or regulations in these areas, Claus Gerhard Bannick believes that the filter crucible would definitely prove a valuable tool in delivering efficient results in fast routine procedures. The microliter filter crucible, fitted with ODW 5, has also already successfully demonstrated its impressive performance in homogeneity testing of reference materials. Indeed, Ulrike Braun's team used the microliter filter crucible to perform the homogeneity control of the materials for a major ring trial undertaken by the European Commission's science and knowledge service (JRC Joint Research Center), which involved a laboratory comparison with simulated bottled water within the scope of a European initiative. "It really worked perfectly," comments the researcher, who therefore also sees an important future application area for

the microliter filter crucible in control tests of this nature. Based on her assessment, the crucible also has great potential for air testing. In contrast to TED-GC/MS, spectroscopic methods cannot be used for analyzing tire abrasion. Ulrike Braun's team has already detected and quantified microplastic particles and tire abrasion particles at the same time in real samples using TED-GC/MS – although not yet with the microliter filter crucible. Thanks to improved measurement accuracy and greater speed, however, she believes that it will deliver important added value for further research projects undertaken within the scope of the regulations on particulate pollution to determine the respective proportions of soot and tire abrasion within fine particulate pollution.

The crucible is currently designed for the TGA of a special provider in TED-GC/MS. However, Ulrike Braun, Claus Gerhard Bannick, and GKD are already considering starting production based on the same prototype for geometries of thermobalances from other manufacturers. The explicit aim here is to ramp up use of the microliter filter crucible in as many applications as possible in order to attain a broad basis of comparable data through standardized routine analyses. A significant workload reduction in laboratories thanks to use of the microliter filter crucible, and the considerable cost and time savings associated with this, mean that this aim is likely to be achieved sooner rather than later.

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