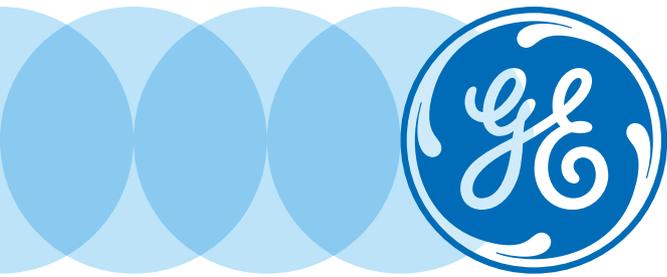


Environmental monitoring – a study using Whatman™ brand filtration products



Introduction

Environmental testing plays an important role in maintaining public health. The ability to detect and characterize substances in air, water, and soil samples is the first step in ensuring that potential risks from these substances are kept below regulated limits. Environmental analysis labs perform a critical function in supporting decision makers who determine what, if any, remediation work is required to clean up sources of contamination. The balance between costly remediation and risk to human health puts tremendous pressure on these labs to deliver accurate, reliable results.

High-quality results depend on several factors. Samples must be collected and handled with care, prepared for analysis in a way that does not compromise their integrity, and analyzed with trustworthy instrumentation. Additionally, the results must be recorded and reported with vigilance. The data presented in this white paper address a single element of environmental testing – filtration as a component of sample preparation. Although filtration is a seemingly small part of a large workflow, the impact of choosing the right filter for a given application can be substantial. The confidence that filtration consumables are not contributing to errors that require rework or root-cause investigations saves money and provides peace of mind. The analyses presented in this white paper will contribute to the informed selection of filters for suspended solids testing of water, preparation of samples prior to chromatographic or colorimetric analysis, and particulate testing of air. Beyond these applications GE Healthcare's Life Sciences business is proud to provide our customers with live technical support for filter selection. Contact information for our trained staff of scientists is provided at the end of the paper.

Water testing – solids analysis

Overview

A key measure of water quality is the concentration and composition of the particulate matter that it contains. Physical matter can be assessed in a number of different ways, depending on the specific health or safety risk being measured or the nature of the sample being assessed. "Total solids" measurement can be subdivided into "total suspended solids" and "total dissolved solids" components. For example, drinking water is often tested for total dissolved solids, because dissolved solids can affect the water taste. Additionally, high levels of dissolved solids can suggest that further analysis is required to assess health risk. High levels of dissolved components can also create process issues in industrial water use.

Total suspended solids is another component of interest, especially in the context of effluent waters. Suspended solids can be assessed by several different methods, but gravimetric assessment of collected material is perhaps the most common. In addition to assessing total suspended matter, determining the fraction of that matter that is volatile may also be required. This process involves heating the sample to remove volatile matter and calculating the result as a proportion of the initial weight of material. The mass lost through the heating step at 550°C is used as an approximation of the amount of organic matter present in the wastewater sample.

Testing of Whatman GF/C™ filters from GE

Introduction

EN 872 is a European standard for determining suspended solids in water, including waste water and effluent. For filters to be used in this method, EN 872 requires:

- that the filter should be borosilicate glass fiber
- that the filter shall not contain any binders
- that the filter shall comprise a mass per unit area of between 50 g/m² and 100 g/m²
- that the filter should exhibit less than 0.017 mg/cm² weight loss when a blank assessment is performed."

Per these requirements, Whatman Grade GF/C filters are manufactured from borosilicate glass microfiber without added binders. They meet the EN 872 requirement of mass per unit area between 50 g/m² and 100 g/m².

EN 872 also describes recommended methods for preparing the filters prior to use. The following test was performed to demonstrate compliance with the method when these preparatory steps are followed.

Methods and results

Whatman Grade GF/C filters (GE) were assessed according to BS EN872:2005 (1), to determine whether the initial weight loss of filters out of the box meets the EN 872 requirement of mass loss less than 0.017 mg/cm².

Three sets of ten 47 mm diameter filters from the same lot were subjected to a blank assessment. Results indicated that the filters require preparation prior to use as defined in EN 872 (i.e., prewash/soak; Fig 1). These three sets were therefore subjected to a washing procedure. Briefly, each GF/C filter was washed with 150 ml of AnalaR NORMAPUR™ water (VWR) under vacuum then dried at 105°C±2°C for 15 hours. This wash was repeated once for sample sets 1 and 2.

Calculations for the 47 mm diameter filters provided an EN 872 limit of 0.294 mg per filter. As shown in Figure 1, all three sets of GF/C filters exhibited < 0.3 mg loss in mass after the first wash, which is less than the 0.017 mg/cm² specified by EN 872.

To test whether variations in humidity would affect GF/C suitability for EN 872, Grade GF/C filters (47 mm) were further subjected to 75% relative humidity (RH) over 72 hours. Filters were tested either directly from the box or after one wash according to the BS EN872:2005 method (1) for blank assessments.

The results are provided in Figure 2.

Conclusion

Based on a mass loss well below the EN 872 requirement after one washing and a mass change less than 0.125 mg in high humidity, Whatman Grade GF/C filters are suitable for use in total suspended solids analysis, regardless of the humidity level.

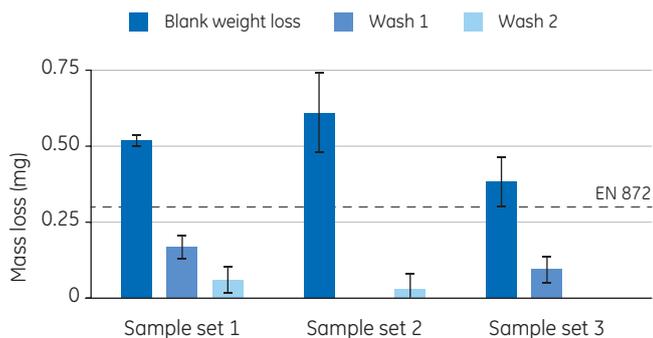


Fig 1. Mass loss of Whatman GF/C filters out of the box and after washing according to EN 872. n = 10 with error bars representing standard deviation.

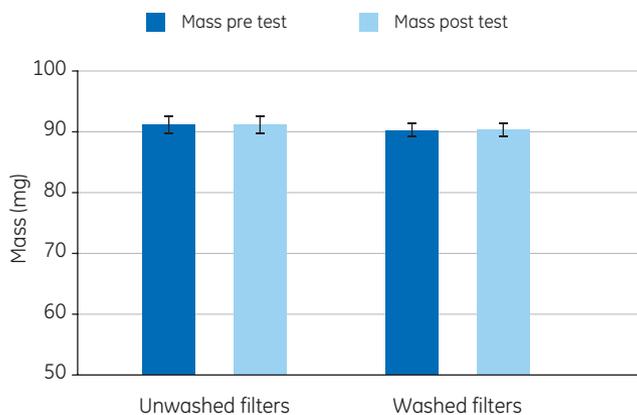


Fig 2. Mass of GF/C filters after 72 h at 75% RH. n = 10 with error bars representing standard deviation.

Testing of Whatman brand 934-AH™ RTU filters

Introduction

Standard method 2540 for the analysis of solids in water and wastewater is subdivided into several sections covering different classes of solids. Parts D and E relate to “total suspended solids” and “fixed and volatile solids,” respectively and use glass fiber filters as part of the analytical method (2). Standard method 2540D specifies the type of filter to be used and the preparative steps that are required in order for the filter to be suitable for use in the method. 934-AH RTU is provided for use straight from the box in Standard method 2540D without further preparation or initial weighing prior to suspended solids testing (Fig 3).

For users wishing to perform volatile suspended solids assessment in Standard Method 2540E, the filter must be heated at 550°C before recording its initial weight.

Methods and results

934-AH RTU filters (GE) were subjected to a high temperature to determine whether any volatile substances were added to the binderless glass fiber during the manufacturing process. Three sets of ten 934-AH RTU filters (42 mm diameter) were fired in a kiln for at least 1 hour at 500°C. All filters were from a single production lot.

As shown in Figure 4, the average mass change was < 1 mg for all three sets of 934-AH RTU filters.

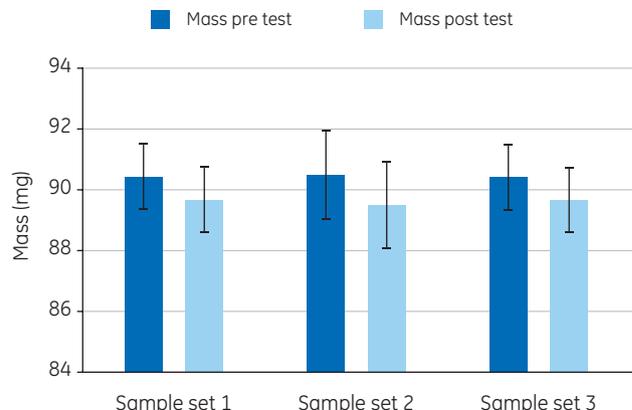


Fig 4. Effect of firing 934-AH RTU 42 mm filters for at least 1 hour at 500°C. n = 10 with error bars representing standard deviation.

Conclusion

934-AH RTU filters conform to the requirements of standard method 2540D, which eliminates the need to pre-wash and pre-weigh. The minimal mass change of 934-AH RTU after heating to 500°C indicates that the filters have a low content of volatiles, which is a consideration for a user performing ignition testing for volatile solids.

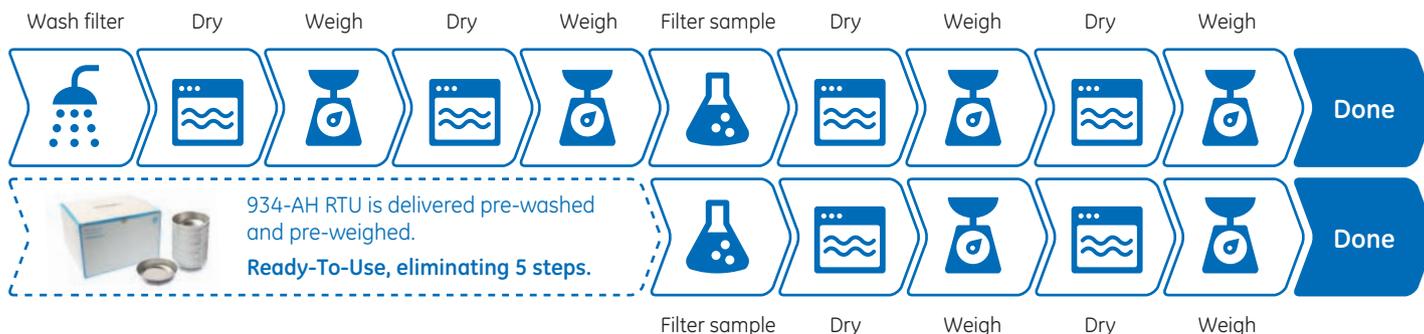


Fig 3. Method 2540D: 934-AH RTU (bottom) vs traditional glass fiber filters (top).

Preparation of samples prior to chromatographic or colorimetric analysis

Overview

Analytical techniques such as HPLC and colorimetric determination are used for quantifying a wide range of chemical species in assessing specific environmental contaminants. Particularly common are analyses to determine aromatic organic compounds including polycyclic aromatic hydrocarbons (PAH), polychlorinated biphenyls (PCB), and inorganic components such as specific metals. Many of the analytical methods used will necessitate some form of sample preparation, very often filtration, to remove physical debris. Sample filtration may be required to increase sensitivity of the assay or to reduce potential damage to instrumentation and analytical components. Regardless of the purpose for filtration prior to analysis, the choice of filter can be crucial. It is important that the chosen filter does not interact with the sample and that it does not contribute to the levels of the compounds of interest that are being detected, either by binding analyte material or releasing extractable materials that can interfere with detection. The level of extractable interference depends on (a) the solvent being filtered, because different solvents will extract different compounds from a membrane, and (b) the optical wavelengths being considered for detection, because a given extractable may not interfere to the same extent at different wavelengths.

Syringe filter extractables data

The study was designed to evaluate the level of extractable interference found in four common solvents at two wavelengths commonly used for organic hydrocarbon analysis in environmental testing.

Methods and results

GE's Whatman GD/X™ syringe filters with nylon, polyvinylidene difluoride (PVDF), and regenerated cellulose (RC) membranes were used in this study. A 3 ml volume of acetonitrile, methanol, water/acetonitrile (60/40), or water/methanol (60/40) was processed through each GD/X syringe filter using a glass syringe. Each filtered sample was collected in HPLC-certified glass vials in 1 ml increments. Following good laboratory practice, the first ml was discarded. After discarding the first 1 ml, a second and a third 1 ml aliquot were filtered. A 100 µl volume of the second and third aliquot was then sampled and analyzed in a spectrophotometer at 254 nm and 280 nm wavelengths.

The absorbance values for all three membrane types at both wavelengths are shown in the following figures.

Conclusion

The data in Figures 5–8 demonstrate low extractable levels for the Whatman GD/X filters with all three membrane types. The RC filters in particular have very low levels

of potential interference at wavelengths relevant to environmental testing. Note that the decision of which membrane to use ultimately depends on the sample solvent, the goals of the filtration, and potentially other chemically relevant factors.

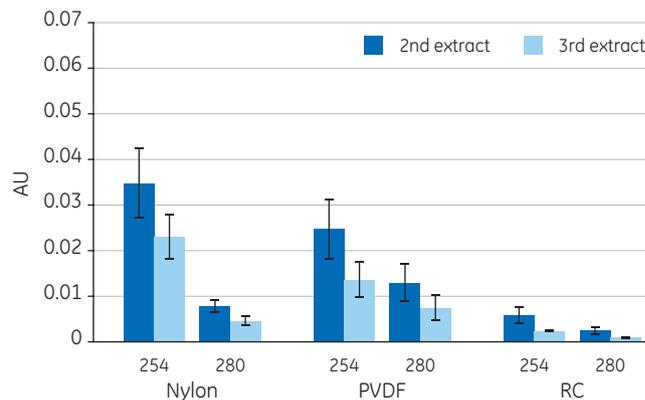


Fig 5. Extractables data for GD/X filters loaded with 3 ml of acetonitrile. The filtrate was collected in three 1 ml aliquots. The first ml was discarded. n = 5 with error bars representing standard deviation.

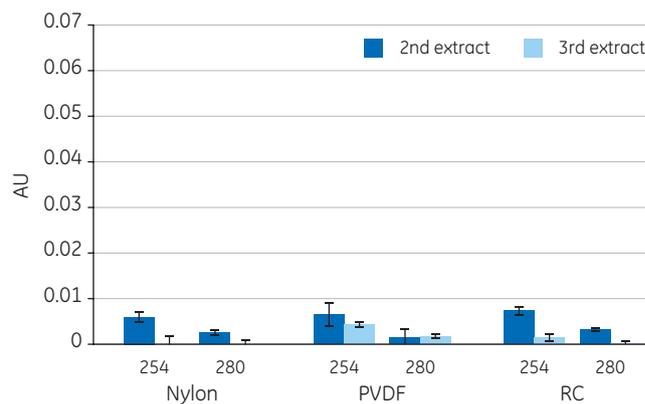


Fig 6. Extractables data for GD/X filters loaded with 3 ml of methanol. The filtrate was collected in three 1 ml aliquots. The first ml was discarded. n = 5 with error bars representing standard deviation.

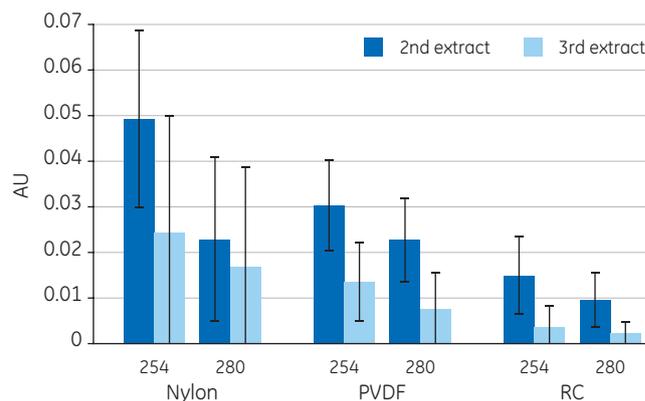


Fig 7. Extractables data for GD/X filters loaded with 3 ml of water/acetonitrile (60/40). The filtrate was collected in three 1 ml aliquots. The first ml was discarded. n = 5 with error bars representing standard deviation.

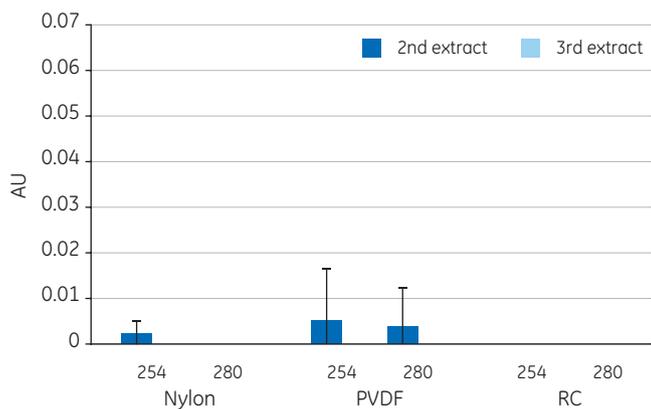


Fig 8. Extractables data for GD/X filters loaded with 3 ml of water/methanol (60/40). The filtrate was collected in three 1 ml aliquots. The first ml was discarded. n = 5 with error bars representing standard deviation.

Particulate loading of syringe filters

Capacity loading

Samples with high levels of particulate matter (e.g., sludges and dissolved sediments) are common in environmental testing. High particulate levels can present challenges during the sample preparation step, such as back pressure build up or the need to use multiple filter units to prepare the volume required for analysis. Under these circumstances, using a filter unit with an integrated prefilter can increase throughput and reduce back pressure. Additionally, use of such a device can potentially eliminate the need for using multiple filter units, which in turn can minimize loss of sample when preparing it for analysis.

Methods and results – capacity loading

Whatman GD/X 0.2 µm nylon syringe filters (GE), which have an integrated multilayer prefiltration media stack, were evaluated in this experiment. In order to challenge the filtration capability, A1 Ultrafine ISO-12103-1 grade test dust at 2, 4, and 6 g/l was prepared in water. The stirred dust particle suspension was fed to a GD/X syringe filter under a constant pressure of 1 bar. The time required to filter each 10 ml increment of sample up to 50 ml was recorded and presented in Figure 9.

Methods and results – sample recovery

Whatman GD/X (GE), which has a stack of media for prefiltration, and Puradisc (GE), which does not have an integrated pre-filter, were used in this study. Corn/water slurries were prepared at 2.5 g/100 ml, 5 g/100 ml, and 10 g/100 ml. The volume of sample that could be recovered before the filter clogged was measured and is shown in Figure 10.

Conclusions – capacity loading

In this study Whatman syringe filters were subjected to flow testing using several particulate loading levels. A1 Ultrafine ISO-12103-1 grade test dust and corn slurry were used to represent application-specific testing. Figure 9 shows that it takes just 8 seconds longer to filter the 6 g/l dust suspension than the 2 g/l suspension. Therefore, GD/X is well-suited for quick filtration even with tough-to-filter samples.

Conclusions – sample recovery

When selecting a syringe filter for environmental testing, it may be important to compare the recovery data for Whatman GD/X, which has a high-efficiency prefiltration media stack, with the data for syringe filters lacking a prefiltration stack. As shown in Figure 10, the volume of sample recovered from GD/X after filtering the 2.5 g/100 ml slurry was 8-fold higher than that recovered from the filter lacking a prefiltration media stack (Puradisc, in this case). The difference was even greater with higher density samples—11-fold higher at 5 g/100 ml and 31-fold higher at 10 g/100 ml.

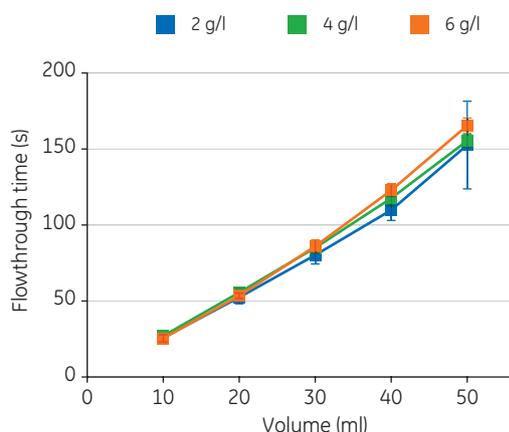


Fig 9. Flowthrough time for three concentrations of dust particle suspensions loaded at constant pressure onto GD/X 0.2 µm nylon syringe filters. n = 5. Error bars represent standard deviation.

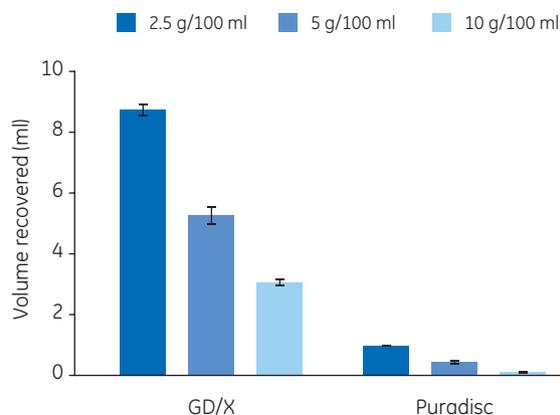


Fig 10. Volume of corn/water slurry samples recovered from Whatman GD/X or Puradisc syringe filters. n = 3. Error bars represent standard deviation.

Air monitoring

Overview

Airborne particulate matter is comprised of a range of particles originating from a variety of different sources. Sampling regimes can differ depending on the origin of the pollutant and the specific particle size range of interest. Sample analysis may assess the levels of physical contaminants present in terms of gravimetric assessment, such as PM 2.5 and PM 10 monitoring. The collected particulate matter can be further assayed to assess the chemical composition and presence of specific ions (e.g., heavy metals) to measure the air quality.

Particulate monitoring

Gravimetric methods are normally used for the determination of particulate matter in the atmosphere. The process involves the collection of airborne particulates by medium- or high-volume air sampling, typically for a minimum of eight hours. The mass of particles is then determined by weighing the filter before and after sampling. The filter that is employed in the collection of the particulate matter must have specific performance characteristics and consistency in order to yield reproducible results. Therefore, care should be given to the choice of the filter medium used. Retention efficiency - the ability to capture and retain a significantly high fraction of airborne particles on the filter membrane - is the most important performance characteristic in ensuring the right choice.

Physical analysis of particulates

Whatman GF/A, EPM 2000, and QM-A filters (all from GE) were evaluated in this study. Dioctylphthalate (DOP) testing was performed with a 0.3 µm challenge particle at a flow rate of 32 l/min. The data are presented in Figure 11.

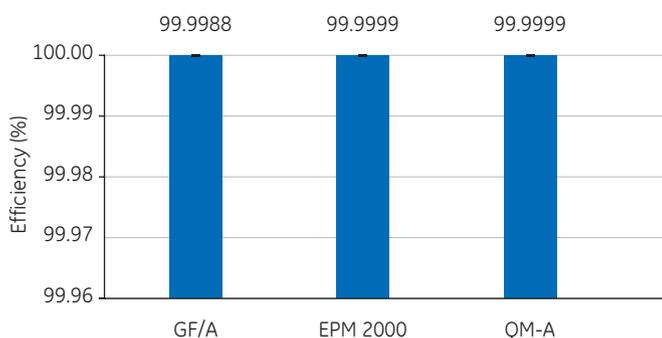


Fig 11. Typical performance of GF-A, EPM 2000 and QM-A glass fiber filters. n = 16 for GF/A, 12 for EPM 2000, and 6 for QM-A with error bars representing standard deviation.

Chemical analysis

Some applications go beyond basic particulate concentration in air to determining the chemical composition of the captured particulates. For quantitative determination of heavy metals, for example, it is important that the filter

chosen should give minimal or no background level for the elements and/or compounds being analyzed and cause minimal interference in the determination. The metal elements of major interest in air pollution monitoring include the heavy metals cadmium (Cd), copper (Cu), lead (Pb), nickel (Ni), manganese (Mn), zinc (Zn), cobalt (Co), and iron (Fe). Table 1 provides the background levels of these and other analytes of interest in Whatman QM-A, EPM 2000, and GF/A air testing filters from GE.

Table 1. Typical trace element levels for GF-A, EPM 2000, and QM-A*

Trace element	QM-A (ppm)	EPM 2000 (ppm)	GF/A (ppm)
Antimony (Sb)	1	6	1
Arsenic (As)	< 1 [†]	< 1 [†]	5
Beryllium (Be)	< 1 [†]	< 1 [†]	< 1 [†]
Cobalt (Co)	< 1 [†]	1.2	< 1 [†]
Cadmium (Cd)	< 1 [†]	< 1 [†]	< 1 [†]
Chromium (Cr)	3	10	16
Copper (Cu)	< 1 [†]	5	1
Iron (Fe)	47	323	223
Lead (Pb)	< 1 [†]	3	5
Manganese (Mn)	2	20	6
Mercury (Hg)	< 1 [†]	< 1 [†]	< 1 [†]
Nickel (Ni)	1	2	1
Silver (Ag)	< 1 [†]	< 1 [†]	< 1 [†]
Thallium (Tl)	< 1 [†]	< 1 [†]	1
Vanadium (V)	< 1 [†]	2	1
Zinc (Zn)	70	51	25583

* Analysis was via complete acid digestion in a microwave and inductively coupled plasma-mass spectrometry (ICP-MS).

[†] Below detectable limits of the test.

Conclusions for air testing

In the context of air monitoring, key performance characteristics of the chosen filter medium include consistent and reproducible retention efficiency of particulate matter and background chemical purity. The data presented in this document allow users to make an informed choice of appropriate filter media based on the specific needs of their application and method.

The selection of individual glass fiber or quartz filter media will be based on individual requirements of the application, such as retention efficiency, thermal stability, and background chemical profile.

All filters tested in this study had retention efficiencies of > 99.99% capture of 0.3 µm particles at a flow rate of 32 l/min. These results suggest that GF/A, EPM 2000, and GF/A are suitable for collection of particulate matter under ambient conditions when no chemical differentiation of those particulates is required. Based on the data presented, if

both high thermal stability and exceptionally low metal background content are required (for chemical analysis of smoke stack emission, for instance), QM-A quartz microfiber is the recommended filter. For other applications the choice of filter will depend on the specific metals being measured. For example, EPM 2000 or QM-A is recommended when determining zinc levels.

Summary

Environmental analysis laboratories are characterized by a high testing volume and the pressure to punctually deliver reliable results in line with regulatory standards. Therefore, the highest quality labs perform best when they have streamlined processes supported by reliable products. Spurious results stemming from use of a sub-optimal device or consumable lead to repeated work and costly root cause investigations. This white paper presents data supporting one key step of environmental sample preparation, namely filter selection. Section 1 presents key characteristics in support of two common methods (standard method 2540 and EN 872) for determination of total suspended and total volatile solids. Section 2 assesses the levels of UV detectable extracts found at two wavelengths commonly used in

analysis of aromatic hydrocarbons. Section 3 presents the background concentrations of heavy metals found in glass and quartz papers commonly used for particulate analysis of air samples. These analyses show that even for a given type of test there is no single, ideal filtration solution for all circumstances. Instead, the filter chosen depends on a number of factors, such as local regulatory standards, sample solvent, or analyte measured. The filter is one part of an environmental testing workflow, but its quality and reliability has a big impact on the final analysis result. The work published here has been performed to aid environmental testing laboratories in selecting the most appropriate filter for their specific applications.

Acknowledgments

ICP-MS results reproduced by permission of Butterworth Laboratories Limited.

References

1. BS EN 872:2005. Water quality. Determination of suspended solids. Method by filtration through glass fibre filters. BSI, London (2005).
2. Standard Methods for the Examination of Water and Wastewater, 22nd edition. American Public Health Association, American Water Works Association, Water Environment Federation (2012).

Ordering information and filtration consultation

For a consultation on which filter to use for your specific application, visit www.gelifesciences.com/contact.

Glass fiber filters for solids analysis, 100/pack

Grades	GF/C	934-AH	934-AH RTU preweighed, prewashed*
Typical particle retention (µm)**	1.2 µm	1.5 µm	1.5 µm
Diameter (mm)	Product code	Product code	Product code
42.5	1822-042	1827-042	9907-042
47	1822-047	1827-047	9907-047
55	1822-055	1827-055	9907-055
70	1822-070	1827-070	-
90	1822-090	1827-090	9907-090

* Each filter is supplied in an individual aluminum pan

** Particle retention rating at 98% efficiency

GD/X syringe filters (glass fiber prefilter), 25 mm diameter

Membrane type	Nylon	PVDF	PTFE	PP	PES	CA	RC	Quantity
Pore size	Product code	Quantity						
0.2 µm	6870-2502	6872-2502	6874-2502	6878-2502	6876-2502	6880-2502	6887-2502	150/pack
0.2 µm	6871-2502	6873-2502	6875-2502	-	6905-2502	-	-	1500/pack
0.45 µm	6870-2504	6872-2504	6874-2504	6878-2504	6876-2504	6880-2504	6882-2504	150/pack
0.45 µm	6871-2504	6873-2504	6875-2504	6879-2504	6905-2504	6881-2504	6883-2504	1500/pack



Glass fiber filters, circles and sheets

Dimensions	25 mm	37 mm	47 mm	50 mm	90 mm	8 × 10 inches (sheet)	
Membrane type	Product code	Quantity					
GF/A	1820-025	1820-037	1820-047	1820-050	1820-090	1820-866	100/pack
EPM 2000	-	-	1882-047	-	-	1882-866	100/pack

Quartz fiber filters

Product name	Dimensions	Product code	Quantity
QM-A quartz fiber filter	25 mm diam.	1851-025	100/pack
	37 mm diam.	1851-037	100/pack
	47 mm diam.	1851-047	100/pack
	50 mm diam.	1851-050	100/pack
	90 mm diam.	1851-090	100/pack
	8 × 10 inches (sheet)	1851-8866	100/pack

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 Amersham Place
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