METROLOGY for HYDROGEN VEHICLES 2

REPORT:

Filtration efficiency of 5 µm and 0.2 µm filters with PSL particles and hydrogen as carrier gas

Authors:	Jesus Marval, Emma Braysher, Jordan Tompkins, Andrew Brown

Confidentiality:	Public
Submission date:	27.02.2023
Revision:	-

https://www.sintef.no/projectweb/metrohyve-2/

Report A3.3.2			
Funding European Metrology Program for Innovation and Research			**Grant agreement no:** 19ENG04
Project name Metrology for Hydrogen Vehicles 2			**Project short name** MetroHyVe 2
Author(s)			**Pages**
Jesus Marval	NPL	jesus.marval@npl.co.uk	16 pages
Emma Braysher	NPL	emma.braysher@npl.co.uk	
Jordan Tompkins	NPL	jordan.tompkins@npl.co.uk	
Andrew Brown	NPL	andrew.brown@npl.co.uk	

Summary

This report shows the filtration efficiency of 5 µm and 0.2 µm filters when challenged by 200 nm and 300 nm polystyrene latex (PSL) reference particles in hydrogen. The Health and Safety considerations, quality control and procedures to carry out such tests are detailed. The results show that new 5 µm and 0.2 µm capture most of the particles (>99 %) of a hydrogen stream. However, the filters' filtration efficiency was significantly different when using a sample already exposed to the test aerosol for a few minutes. The results show no significant difference between the filtration efficiency of the 5 µm and 0.2 µm filters and the filtration efficiency with 300 and 200 nm PSL particles. For more details about this project please visit https://www.sintef.no/projectweb/metrohyve-2/.

Confidentiality	Public

This project "Metrology for Hydrogen Vehicles 2" (MetroHyVe 2) has received funding from the EMPIR programme co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation programme under Grant agreement No [19ENG04].

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Contents

Introduction

The recent development of fuel cell electrical vehicles (FCEV) has increased significantly following the need of European economy to move toward a net zero economy by 2050.

FCEVs have strict specification for hydrogen fuel quality as the presence of contaminants can impact the lifetime of fuel cells and of the FCEV [1]. Thirteen gaseous contaminants and particulates should be monitored according to international standards ISO 14687 [2] or EN 17124 [3]. The international standard requires maximum particulate concentration to be below 1 mg/kg.

In the recent years, a strong focus was on the gaseous contaminants and the development of the metrological methods to perform these measurements [4]. The particulate analysis was less investigated due to the complexity of the measurement and the sampling equipment [5].

The international standards are not specifying the filter type or pore size. From the recent MetroHyVe 2 good practice guide [6], there is a variety of filters used for this measurement (e.g., Pall TF-200 47mm; Millipore Mitex LSWP04700; HahneMühle PT 020 47 BL). There is no comparison study between the different filters used, even if the efficiency comparison of different types of filters is key to support the standardisation and the reproducibility of this measurement worldwide. One parameter of interest is the filter pore size that vary from 5.0 µm to 0.2 µm. However, there is little information in the literature about the impact of the pore size on the filtration efficiency particulate in hydrogen gas.

The aim of the study was to assess the filtration efficiency of 5 µm and 0.2 µm filters when challenged by polystyrene latex (PSL) reference particles in hydrogen. These results will provide the first study on the impact of filter pore size for filtration efficiency of reference particles in hydrogen matrix. It will support the standardisation effort in particulate sampling from hydrogen refuelling stations.

Test setup

Description

The test setup was designed to introduce a known amount of reference particles into a hydrogen stream. It will create a reference particles hydrogen stream. Then different filters are exposed to this reference particles hydrogen stream at pressure. The particles concentration is measured before and after the filter (using calibrated particle counter) to determine in real time the filter efficiency to retain particles of different sizes. The tests were performed at the National Physical Laboratory (Teddington, TW11 0LW, United Kingdom). Figure 1 shows a schematical representation of the devices used for carrying out the tests. Most of the devices were placed inside a fume hood for safety reasons.

The hydrogen gas was high purity (Hydrogen BIP+, AirProducts, UK). The nitrogen gas was high purity (50L cylinder BIP, AirProducts, UK). The pressure was regulated below 10 bar using two stages pressure regulators (HiQ regulator for nitrogen from BOC, UK and hydrogen regulator from Air Products, UK). The flow rate was set at 2 l min^{-1} by using a and a mass flow model mass view MV-194-H2 (Bronkhorst UK Ltd., UK).

A TOPAS model ATM 210 (Topas GmbH, DE) was used as the aerosol generator. This device can handle an inlet and outlet pressure of up to 15 bar and 10 bar, respectively. The outlet pressure was set at 2 bar. Certified polystyrene latex spheres (PSLs) of a size of either (203 ± 4) nm model 3200A (Thermo Fisher Scientific Inc, USA) or (303 ± 6) nm model 3300A (Thermo Fisher Scientific Inc, USA) were aerosolised to produce the tests aerosol. The TOPAS ATM 210 vessel was filled with around 50 ml of ultrapure water (15 MΩ) and 20 drops of the PSL solution were added to the vessel.

A PALAS model Promos 2000 (Palas GmbH, DE) together with a PALAS model Welas 2200 head (Palas GmbH, DE) was used as the particle spectrometer. This combination provides the particle concentration in terms of optical particle size in the size range of 0.14 – 10 µm. The maximum allowed particle concentration is 1.6×10^4 cm^{-3}.

The PALAS Promos 2000 internal pump has an inlet flow rate of 5 l min^{-1}. The aerosol at 2 l min^{-1} (e.g., hydrogen with PSL particles) passed only through the PALAS Welas 2200 head while the internal pump of PALAS Promos 2000 was sampling clean air from an HEPA filter. Since the flow passing through the Optical Particle Counter (OPC) pump was different than the flow through the OPC head, a correction of the total particle concentration provided by the OPC is needed. In this case, the data must be corrected by a factor of 2.5 (ratio between the two flow rates).

The 4-way valve Swagelok model SS-45YF4 (Swagelok Company, USA) shown on Figure 1 and Figure 4 was used for including or excluding the 47 mm filter holder model 1235 (Pall Corporation, USA) (i.e., the filter under test) from the sampling line. In this way, the particle number concentration (PNC) of the either filtered or unfiltered aerosol was measured, without disturbing the sampling lines.

Figure 2 shows the test setup used during the tests.

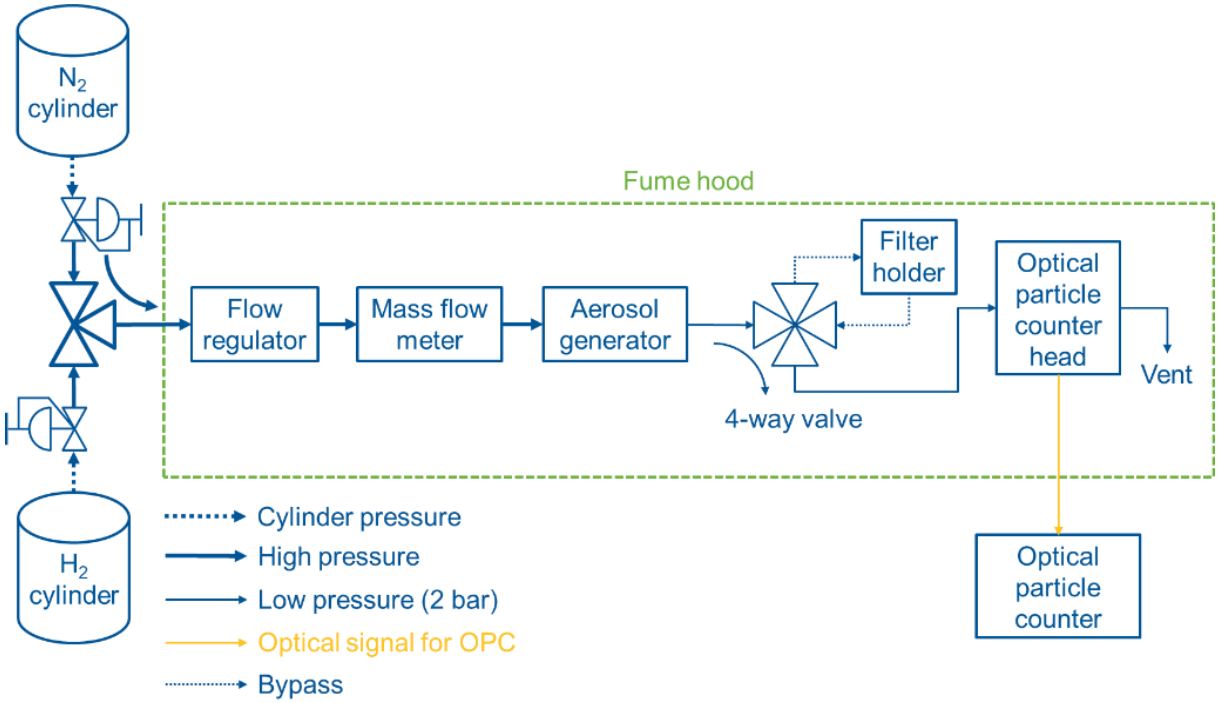

Figure 1. Test setup diagram.

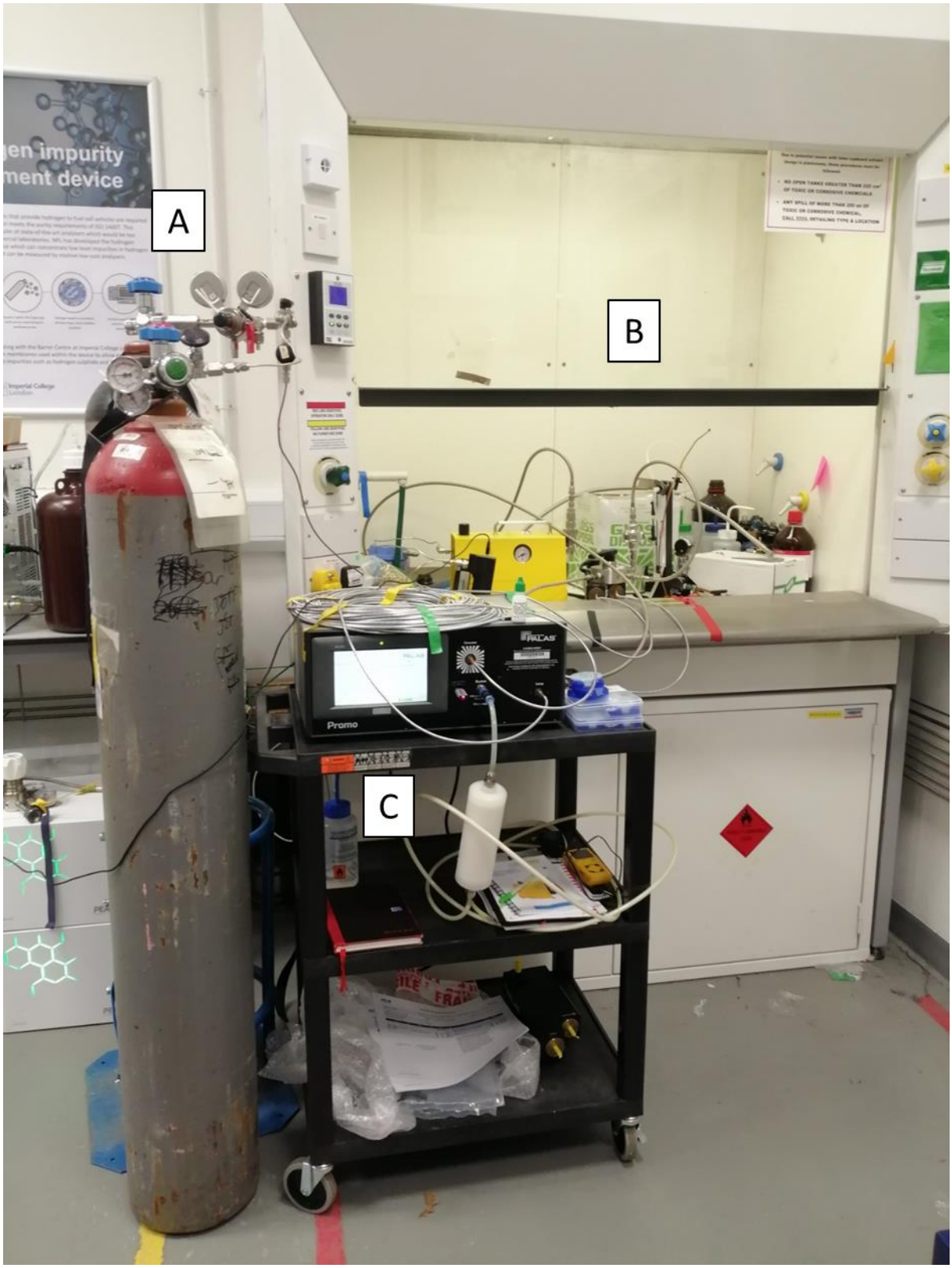

Figure 2. Test setup. A is the gas cylinders with regulators. B is the fume hood with some instruments. C is the optical particle counter with its pump sampling from an HEPA filter.

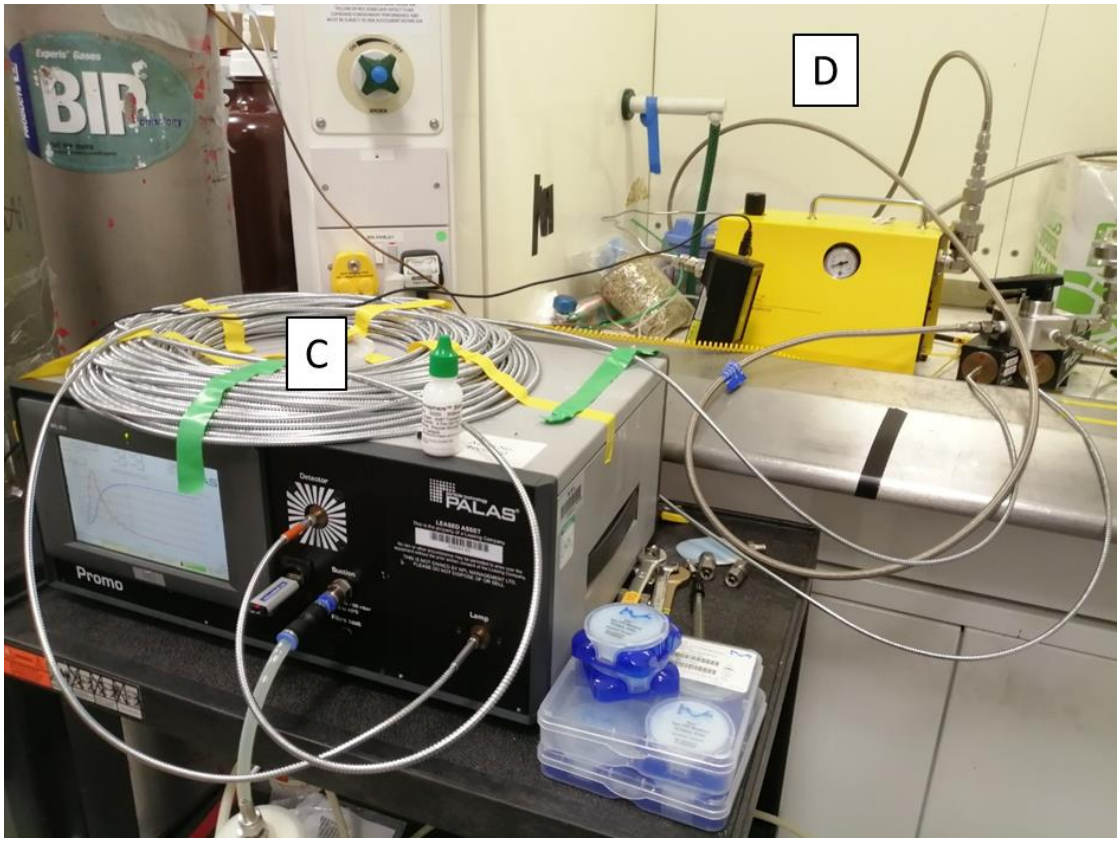

Figure 3. Optical particle counter (C) and aerosol generator (D) used during the tests.

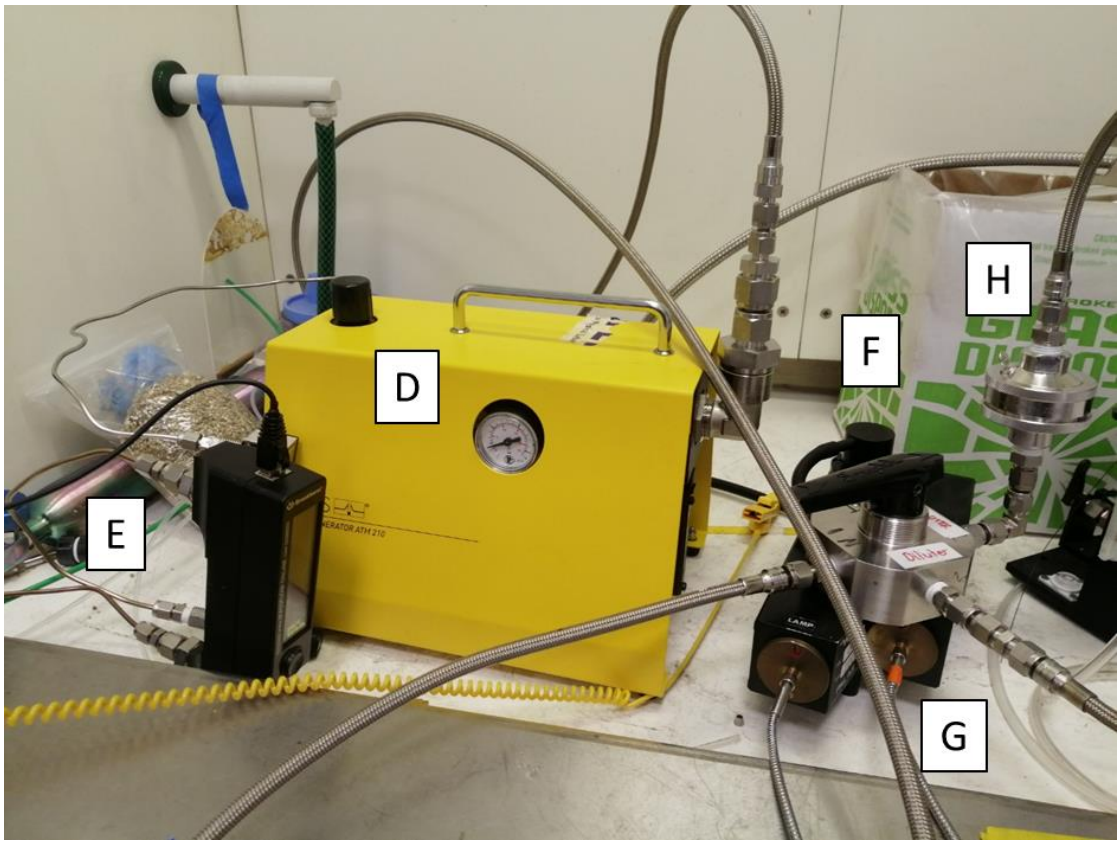

Figure 4. D is the aerosol generator. E is the mass flow meter. F is the 4-way valve. G is the optical particle counter head. H is the filter holder.

Filtration efficiency of 5 µm and 0.2 µm filters with PSL particles and hydrogen as carrier gas

Health and Safety practice

Since hydrogen is highly flammable, a health and safety risk assessment was carried out to understand the risks and reduce them whenever possible.

All the system was grounded. There were no electronics devices at the hydrogen outlet. Therefore, there were no electric ignition sources. Furthermore, nitrogen was used for purging the lines to remove any trace of air in the system before introducing hydrogen gas. A 3-way valve was used to purge the system with nitrogen before switching to the flammable gas. Before the initiation of the experiments, a leak test was performed.

The system was vented into a fume hood with an extraction rate of 30,000 l min^{-1}, away from any potential ignition sources. At a hydrogen flow rate of 2 l min^{-1} diluted in a 30,000 l min^{-1}, a mixture with less than 0.01% hydrogen in air could be produced. The lower flammable limit of hydrogen is 4% and following HSES safety guidelines, the tests were well below the 1% required.

The aerosol generation was carried out in a fume hood and operators were trained in using them. A check of the setup was performed by two operators independently.

Hydrogen and nitrogen sources were gas cylinders. Making and breaking pressurised systems (i.e., gas cylinders and pressure regulators) was conducted only by experienced and trained operators. As making and breaking pressurised setups is a medium risk lone working activity, a *buddy system* was necessary and therefore, an operator informed a buddy before carrying out this task.

Hydrogen can leak through the conductive tube normally used when sampling aerosols. Therefore, the tests were performed using stainless steel tubes to minimise hydrogen leakage.

Palas Promo 2000 (OPC) lab-based calibration

Before each measurement campaign, the particle spectrometer was calibrated by following the manufacturer's procedure [7]. Monodisperse calibration dust "CalDust 1100" was used for the calibration, and the *photomultiplier amplification* and the *measured velocity* were adjusted to 2.520 and 8.527 m s^{-1}, respectively.

Filters tested

The tests were performed using the following filters:

- Fluoropore™ filter reference FGLP04700, batch number R9BA86874 (Merck Millipore Ltd, IRL). The filter is a hydrophobic, polytetrafluoroethylene (PTFE) polymer membrane bonded to a high-density polyethylene support to improve the handling characteristics of the filter for normal applications. The filter has 0.2 µm pore size, 47 mm diameter.
- Mitex™ filter reference LSWP04700, batch number RODB51090 (Merck Millipore Ltd, IRL). The filter is a hydrophobic, polytetrafluoroethylene (PTFE) polymer membrane. The filter has 5.0 µm pore size, 47 mm diameter.
- Hahnemuhle filter reference PTF50047BL, batch number 20201116001 (Hahnemuhle, DE). The filter is made entirely of PTFE (polytetrafluorethylene), reinforced by a Polypropylene net. The filter has 5.0 µm pore size, 47 mm diameter.
- Hahnemuhle filter reference PTO2O47BL, batch number 20201116002 (Hahnemuhle, DE). The filter is made entirely of PTFE (polytetrafluorethylene), reinforced by a Polypropylene net. The filter has 0.2 µm pore size, 47 mm diameter.

- Omnipore filter reference JGWP04700, batch number R1MB56167 (Merck Millipore Ltd, IRL). The filter is a hydrophobic, polytetrafluoroethylene (PTFE) polymer membrane bonded to a high-density polyethylene support to improve the handling characteristics of the filter for normal applications. The filter has 0.2 µm pore size, 47 mm diameter.

Results

Total particle number concentration during the tests

Figure 5 shows the total particle number concentration over time. The region with title "300 nm particles" corresponds to the tests carried out using 300 nm PSL particles. Similarly, the region with title "200 nm particles" corresponds to the tests carried out using 200 nm PSL particles.

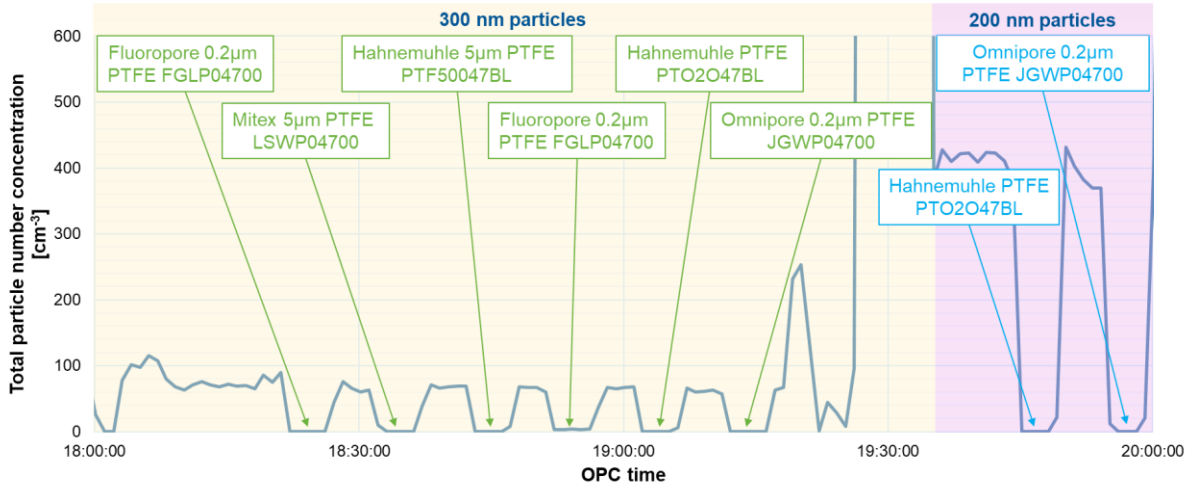

Figure 5. Total particle number concentration as a function of the time. The displayed changes are related to turning the 4-way valve (i.e., including and excluding the filter holder and therefore filtering or not the test aerosol).

300 nm PSL particles (clean filters)

All the filters mentioned above were exposed with an aerosol made-up of 300 nm PSL particles. All the samples tested were clean (i.e., those were not challenged with any aerosol before the tests). Table 1 shows these results.

The average upstream particle number concentration reported by the OPC was 68 particles per cm^3. However, since the actual flow rate passing through the OPC head was 2 l min^{-1} instead of 5 l min^{-1}, the actual particle number concentration was approximately 171 particles per cm^3. Since the filtration efficiency is obtained by the ratio between the upstream and the downstream particle concentration, it was not necessary to correct the particle concentration when calculating the filtration efficiency.

Table 1. Filtration efficiency measured with 300 nm PSL particles.

Filter name	Filtration efficiency
Fluoropore 0.2µm PTFE FGLP04700	99.90%
Mitex 5µm PTFE LSWP04700	99.75%
Hahnemuhle 5µm PTFE PTF50047BL	99.84%
Hahnemuhle 0.2µm PTFE PTO2O47BL	99.97%
Omnipore 0.2µm PTFE JGWP04700	99.95%

200 nm PSL particles (clean filters)

After carrying out the measurements with 300 nm particles, the two filters with the highest filtration efficiency were selected to be tested also with 200 nm PSL particles. All the samples tested were clean (i.e., those were not challenged with any aerosol before the tests). Table 2 shows these results.

The average upstream particle number concentration was 404 particles per cm^3. However, since the actual flow rate passing through the OPC head was 2 l min^{-1} instead of 5 l min^{-1}, the actual particle number concentration was approximately 1010 particles per cm^3. Since the filtration efficiency is obtained by the ratio between the upstream and the downstream particle concentration, it was not necessary to correct the particle concentration when calculating the filtration efficiency.

Table 2. Filtration efficiency measured with 300 nm PSL particles.

Filter name	Filtration efficiency
Hahnemuhle 0.2µm PTFE PTO2O47BL	99.98%
Omnipore 0.2µm PTFE JGWP04700	99.96%

Assessing the effect of aerosol exposure into filtration efficiency

The impact of the test aerosol on the filtration efficiency was assessed. These measurements were carried out with 300 nm PSL particles on the filter *Fluoropore 0.2 µm PTFE FGLP04700*. First, the filtration efficiency of a brand-new filter was measured. Second, its filtration efficiency was measured again after already being exposed for 5 min to the challenging aerosol.

Table 3. Filtration efficiency measured with 300 nm PSL particles of the filter Fluoropore 0.2µm PTFE FGLP04700.

Filter condition	Filtration efficiency
New	99.90%
After 5 min exposure	94.48%

The results show a sharp decrease in the filtration efficiency after exposing the sample to a few minutes of the challenging aerosol.

Uncertainty calculations

The filtration efficiency was calculated using Eq. 1.

$$E\ [\%] = 100\left(1 - \frac{D}{U}\right) \qquad \textit{Eq. 1}$$

Where:

E = Filtration efficiency
D = Downstream total particle concentration (i.e., total particle concentration of the filtered aerosol)
U = Upstream total particle concentration (i.e., total particle concentration of the unfiltered aerosol)

Table 4 shows the values used to calculate the filtration efficiency of each filter.

Since most of the tested filters have a filtration efficiency close to 100%, the downstream concentrations were very low. Consequently, the mean and standard deviation of the downstream measured values were often quite similar between them. Therefore, if a Gaussian distribution were assigned to the downstream concentration, there is a significant chance that it will provide negative particle concentration values, which is unphysical. For that reason, we concluded that it is not sensible

to use the standard analytical approach for uncertainty evaluation, and a numerical approach must be used.

Table 4. Average and standard deviation of the total particle concentration during the tests.

Row Labels	Average Total PNC [cm^{-3}]	StdDev Total PNC [cm^{-3}]	Relative StdDev
(03) Upstream PNC 300 nm	71.51	7.83	11%
(03) Fluoropore 0.2µm PTFE FGLP04700	0.07	0.07	105%
(04) Upstream PNC 300 nm	67.64	4.44	7%
(04) Mitex 5µm PTFE LSWP04700	0.17	0.07	41%
(05) Upstream PNC 300 nm	67.22	3.15	5%
(05) Hahnemuhle PTFE PTF50047BL	0.11	0.08	77%
(06) Upstream PNC 300 nm	66.22	2.78	4%
(06) Fluoropore 0.2µm PTFE FGLP04700	3.66	0.62	17%
(07) Upstream PNC 300 nm	64.76	3.08	5%
(07) Hahnemuhle PTFE PTO2O47BL	0.02	0.04	200%
(08) Upstream PNC 300 nm	62.51	2.65	4%
(08) Omnipore 0.2µm PTFE JGWP04700	0.03	0.08	224%
(09) Upstream PNC 200 nm	404.26	21.89	5%
(09) Hahnemuhle PTFE PTO2O47BL	0.09	0.09	100%
(10) Upstream PNC 200 nm	390.94	26.35	7%
(10) Omnipore 0.2µm PTFE JGWP04700	0.14	0.13	92%

An exponential distribution was assigned to the downstream concentration, except to the exposed "Fluoropore 0.2 µm PTFE R9BA86874" test where a Gaussian distribution was used as in this case the relative standard deviation of the downstream concentration was much lower than in the other cases. On the other hand, a Gaussian distribution was assigned to the upstream concentration. Therefore, a Monte Carlo approach was adopted for estimating the total expanded uncertainty with a 95% coverage interval. Since the exponential distribution is asymmetrical around the mean, the resulted total expanded uncertainty would be asymmetrical as well.

For each test, the mean and standard deviation data in Table 4 was fed to a thoroughly tested Monte Carlo simulation routine on MATLAB and 100,000,000 simulations were performed.

A convergence approach was adopted, and results were accepted only if the calculations of all the mean, the standard deviation, and the confidence intervals have stabilised to two significant figures.

Using the above approach, the total expanded uncertainty with a 95% coverage interval of each test was estimated. The average difference between the filtration efficiencies in Table 4 and the ones obtained with the Monte Carlo approach was 0.0018%. The results with their associated uncertainty are shown in Table 5.

Table 5. Filtration efficiency results with associated uncertainty.

Filter name	Particle size [nm]	Filter condition	Filtration efficiency	*Relative expanded uncertainty*
Fluoropore 0.2µm PTFE FGLP04700	300	Clean	99.90%	–0.20% +0.10%
Mitex 5µm PTFE LSWP04700	300	Clean	99.75%	–0.51% +0.25%
Hahnemuhle 5µm PTFE PTF50047BL	300	Clean	99.84%	–0.32% +0.16%
Fluoropore 0.2µm PTFE FGLP04700	300	Exposed	94.48%	–2.00% +2.00%
Hahnemuhle 0.2µm PTFE PTO2O47BL	300	Clean	99.97%	–0.07% +0.03%
Omnipore 0.2µm PTFE JGWP04700	300	Clean	99.95%	–0.11% +0.05%
Hahnemuhle 0.2µm PTFE PTO2O47BL	200	Clean	99.98%	–0.04% +0.02%
Omnipore 0.2µm PTFE JGWP04700	200	Clean	99.96%	–0.07% +0.04%

Significance analysis

Filtration efficiency of 5 µm and 0.2µm filters with 300 nm PSL particles

Figure 6 shows the significance analysis between the tests with 5 and 0.2 µm filters when challenged with 300 nm PSL particles. For the 5.0 µm filters, we used the average of filters “Mitex 5µm PTFE RODB51090” and “Hahnemuhle 5µm PTFE PTF50047BL”. Similarly, for the 0.2 µm filters, we used the average of filters “Fluoropore 0.2 µm PTFE FGLP04700”, “Hahnemuhle 0.2 µm PTFE PTO2O47BL”, and “Omnipore 0.2 µm PTFE JGWP04700”.

Although the results showed a higher filtration efficiency for 0.2 µm filters, no significant difference was found between 5 and 0.2 µm filters with a confidence level of 95%.

These results correspond only to the tests performed on the clean filters (i.e., those not challenged with any aerosol before the tests).

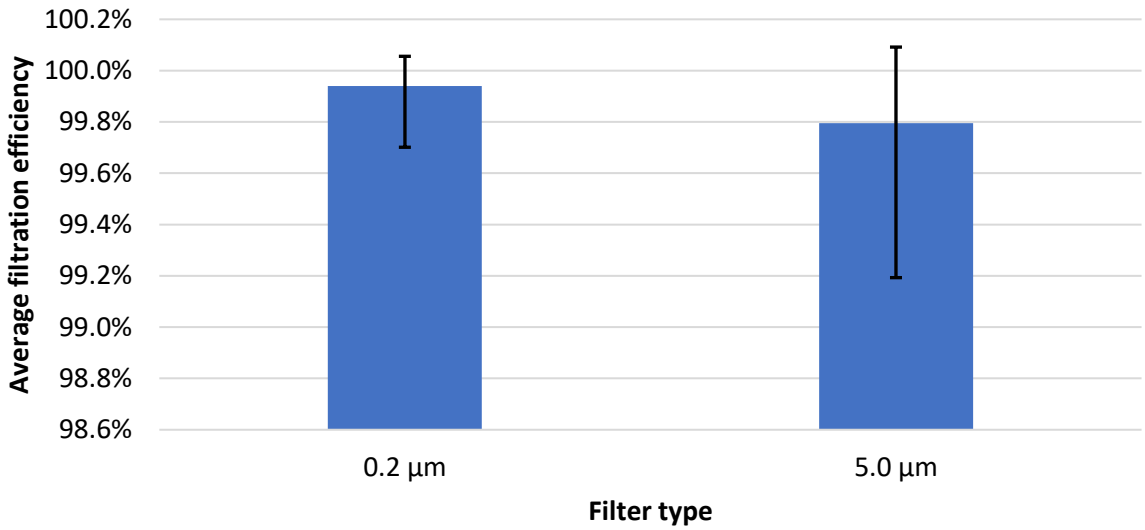

Figure 6. Significance analysis of 5 µm and 0.2 µm filters when challenged with 300 nm PSL particles (the error bars are the summation in quadrature of the uncertainties in Table 5). For the 5.0 µm filters, we used the average of filters "Mitex 5 µm PTFE RODB51090" and "Hahnemuhle 5 µm PTFE PTF50047BL". For the 0.2 µm filters, we used the average of filters "Fluoropore 0.2 µm PTFE FGLP04700", "Hahnemuhle 0.2 µm PTFE PTO2O47BL", and "Omnipore 0.2 µm PTFE JGWP04700"

Filtration efficiency with 300 and 200 nm PSL particles

Figure 7 shows the significance analysis between 300 and 200 nm PSL particles tests. These tests were performed only on filters "Hahnemuhle 0.2 µm PTFE PTO2O47BL" and "Omnipore 0.2 µm PTFE JGWP04700". Although the results showed a slightly higher filtration efficiency with 200 nm PSL particles, no significant difference was found between 300 and 200 nm PSL particles with a confidence level of 95%.

The relative expanded uncertainty of the measurements with 200 nm PSL particles is smaller than those with 300 nm PSL particles. This behaviour could be related to the total upstream particle concentration that was higher with the tests with 200 nm PSL particles. Therefore, the higher the total particle number concentration, the more stable the aerosol and consequently, the lower the expanded uncertainty.

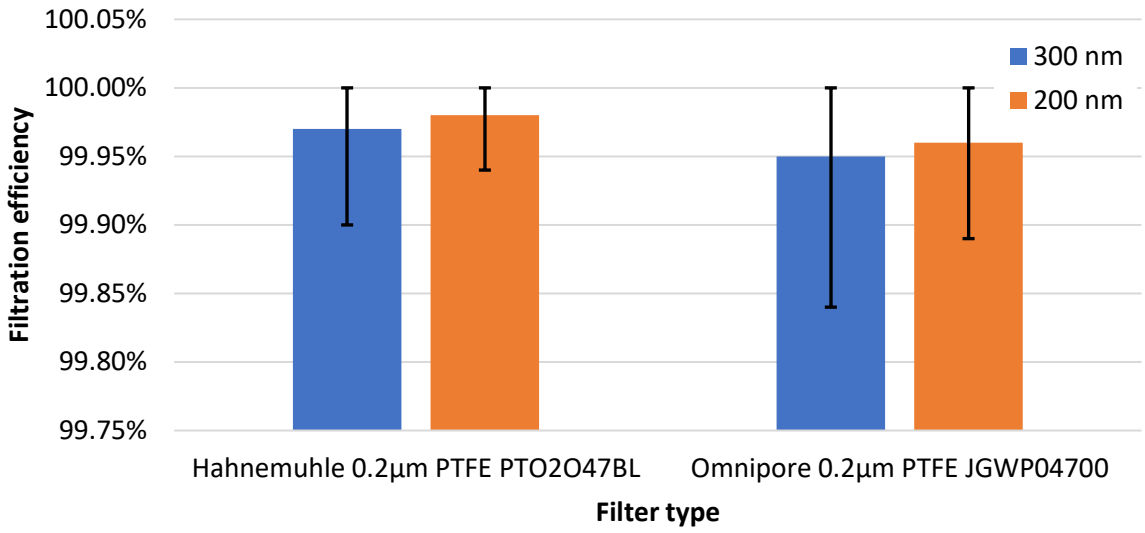

Figure 7. Significance analysis of filtration efficiency with 300 and 200 nm particles (the error bars are the expanded uncertainty of the results in Table 5).

Filtration efficiency of a clean and exposed filter

Figure 8 shows the significance analysis between the tests on a clean and already exposed filter. These tests were performed only on filter "Fluoropore 0.2 µm PTFE R9BA86874". The results showed a higher filtration efficiency on the clean filter, and a significant difference was found between the clean and already exposed filter with a confidence level of 95%.

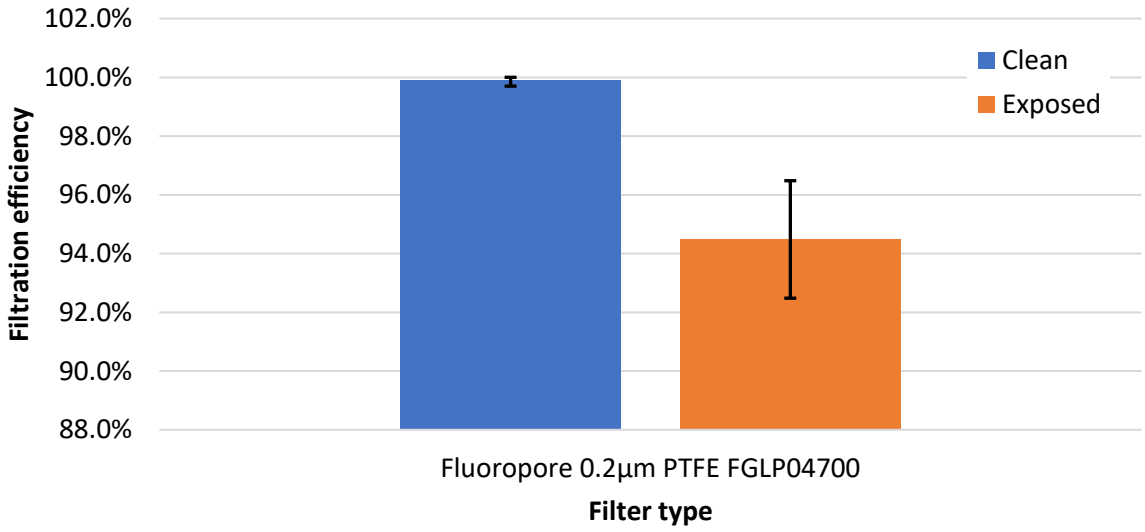

Figure 8. Significance analysis of filtration efficiency of the clean and exposed filter (the error bars are the expanded uncertainty of the results in Table 5).

Conclusion

This report aimed to show how to assess the filtration efficiency of 5 µm and 0.2 µm filters when challenged by PSL reference particles of 200 and 300 nm in hydrogen.

The results showed that new 5 µm and 0.2 µm pore size filters both capture > 99 % of the particles of a hydrogen stream. The results do not provide a significant difference between the filtration efficiency

of the 5 µm and 0.2 µm filters and the filtration efficiency with 300 and 200 nm PSL particles. The filtration efficiency's relative expanded uncertainties of the tests on the new filters were small confirming the high filtration efficiency values.

However, the filtration efficiency decreased when the filter has been already exposed to the test aerosol for a few minutes. This decrease was assessed only with filter "Fluoropore 0.2 µm PTFE R9BA86874" and this test was not carried out with any other filter type.

Suggestions for future studies

Filtration efficiency measuring protocol

During our study, the filtration efficiency was calculated by measuring the upstream and downstream particle concentrations for a period of 5 min once per filter. Therefore, only a single value of filtration efficiency per filter was calculated.

For future studies, we suggest measuring several times both the upstream and downstream particle concentration, starting from the upstream particle concentration, and finishing again with the upstream particle concentration. In this way, one could better calculate the filtration efficiency by using a value of the downstream particle concentration and the two values of the upstream particle concentration, the upstream before and upstream after the said downstream measurement.

This approach would allow one to consider any bias in the upstream particle concentration during the tests and therefore, will improve the reliability and confidence of the filtration efficiency values obtained. However, a disadvantage of this approach is that a given filter will be exposed for a longer time to the aerosol, and this could affect its performance.

Repeatability of each filter type

Only one sample of each filter type was used to determine the filtration efficiency.

For future studies, we suggest testing at least three samples of each filter type. This would allow one to assess any potential difference within batches given filter type.

Re-exposure tests

The impact of the challenging aerosol on the filtration efficiency was assessed only for filter "Fluoropore 0.2 µm PTFE R9BA86874". In this case, the filtration efficiency was reduced by 5.43%, after having exposed the filter sample to the challenging aerosol for 5 min.

For future studies, we suggest carrying out this test on each filter type tested and additionally, assessing the impact of the exposure time on the filtration efficiency by exposing the filter to the challenging aerosol for different periods (e.g., 5 min exposure, 10 min exposure, 20 min exposure).

Pressure and flow

The particulate sampling is currently realised at high pressure (up to 70 MPa) and high flow (up to 20 g s^{-1}). The impact of particles with high velocity or high pressure is important to evaluate especially in term of filter damage, degradation. The current experiments were performed at low pressure and low flow. It is important to consider how to extrapolate or expand facility to realise experiments closer to the operations conditions.

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