



## Inter-Laboratory Validation of the Method to Determine the Filtration Efficiency for Airborne Particles in the 3–500 nm Range and Results Sensitivity Analysis

Panagiota Sachinidou<sup>1,2</sup>, Yeon Kyoung Bahk<sup>1,2</sup>, Min Tang<sup>3</sup>, Ningning Zhang<sup>3</sup>,  
Shawn S.C. Chen<sup>3</sup>, David Y.H. Pui<sup>3</sup>, Bruno Araújo Lima<sup>4</sup>, Gabriele Bosco<sup>4</sup>,  
Paolo Tronville<sup>4</sup>, Thomas Mosimann<sup>5</sup>, Mikael Eriksson<sup>6</sup>, Jing Wang<sup>1,2\*</sup>

<sup>1</sup> *Laboratory of Advanced Analytical Technologies, Empa, 8600 Dübendorf, Switzerland*

<sup>2</sup> *Institute of Environmental Engineering, ETH Zurich, 8093 Zurich, Switzerland*

<sup>3</sup> *Particle Technology Laboratory, Department of Mechanical Engineering, University of Minnesota, Minneapolis, MN 55414, USA*

<sup>4</sup> *Department of Energy, Politecnico di Torino, 10129 Torino, Italy*

<sup>5</sup> *Unifil AG, Filbertechnik, CH-5702 Niederlenz, Switzerland*

<sup>6</sup> *Camfil Svenska AB, SE-619 33 TROSA, Sweden*

---

### ABSTRACT

The filtration of airborne nanoparticles is becoming an important issue as they are produced in large quantities from material synthesis and combustion emission. Current international standards dealing with efficiency test for filters and filter media focus on measurement of the minimum efficiency at the most penetrating particle size. The available knowledge and instruments provide a solid base for development of test methods to determine the effectiveness of filtration media for airborne nanoparticles down to a single-digit nanometer range.

An inter-laboratory evaluation is performed under the Technical Committee 195 of European Committee for Standardization (CEN/TC195) for the development of the methodology to determine effectiveness of filtration media for airborne particles in the 3–500 nm range. Statistical analysis of the results was performed according to ISO 5725-2 in order to evaluate the test procedure and sensitivity analysis was carried out to identify the factors that could possibly affect the test results.

Inter-laboratory analysis revealed some deviation among the experimental results. The statistical analysis showed a less than 20% deviation. This deviation could be attributed to the difference among the experimental setups used by the laboratories. The sensitivity analyses did not indicate a strong influence by the temperature, relative humidity, flow distribution, challenging particle concentration, or particle density on the filtration efficiency in the parameter ranges used in the inter-laboratory test. However, the charging status of the filter affected the filtration efficiency.

**Keywords:** Filtration efficiency; Inter-laboratory tests; Sensitivity analysis; Statistical analysis.

---

### INTRODUCTION

Filtration of airborne nanoparticles is crucial due to the increased produced quantities from material synthesis and combustion emissions (Wang and Tronville, 2014). More small particles are being produced compared to the past due to the blossom of the field of nanotechnology. Many experimental and theoretical studies for particles down to single digit nanometers have already been performed by many researchers such as Kim *et al.* (2009), Wang *et al.*

(2007), Thomas *et al.* (2013), Huang *et al.* (2007) and Steffens and Coury (2007a, b).

Filtration testing is very challenging because many parameters can affect the filtration efficiency. Sachinidou *et al.* (2017) concluded that particle size distribution and charge could possibly affect the filtration test accuracy. Kim *et al.* (2006) and Yang and Lee (2004) showed that relative humidity did not influence the filtration efficiency. However, the charge status of the filter media could affect the filtration efficiency as stated by Brown (1993), Lore *et al.* (2011). Huang *et al.* (2007) and Maze *et al.* (2007) showed that flow temperature can alter filtration efficiency. Thus, it is crucial to determine a reliable procedure for the filtration test which could minimize the artifacts. Even though there are a number of standards for testing air filters that cover a large particle size range like ASHRAE

---

\* Corresponding author.

Tel.: +41 44 633 36 21

E-mail address: jing.wang@ifu.baug.ethz.ch

52.2-2017, EN 1822:2009, EN 779:2012, ISO 16890:2016 ISO 29461-1:2013, and ISO 29463-3:2011 up to several micrometers, there is no standard focusing on the filtration of nanoparticles down to single digit nanometers. It is very challenging to develop such a procedure.

After the development of a procedure to test the filtration efficiency for nanoparticles down to single digit nanometers, inter-laboratory testing should be performed so as to evaluate the reliability of the test method and statistical tools should be applied to analyze the results.

In order to evaluate the procedure to test the filter filtration efficiency for airborne particles in the size range of 3–500 nm, five different laboratories designated randomly as A, B, C, D and E carried out the same experiments, the so-called round robin tests. A qualification procedure for the test rig and apparatus was performed before the round robin tests by each lab in order to exclude systematic errors. Repeatability and reproducibility of the test procedure were evaluated with statistical analysis according to ISO 5725-2.

A sensitivity study was also performed. Part of this study was based on the round robin test results. The aim of the study is to reveal what parameters could affect the filtration efficiency and possibly explain the deviation among the experimental data reported in the round robin test. In addition, the results are important for the specification of the range of the parameters in the test method. Relative humidity, temperature, flow distribution upstream the filter holder, upstream particle concentration are several parameters that can affect the measured filtration efficiency. Furthermore, the challenging particle size distribution, neutralization efficiency, sheath to aerosol flow ratio (SAFR) in the particle classifier could cause measurement artifacts which can contribute to the deviation in the experimental results as Sachinidou *et al.* (2017) stated.

## FILTRATION EFFICIENCY TESTS

The test system for filtration efficiency of airborne particles usually involves an aerosol generation part, particle measurement part, filter holder system, and other parts for pressure measurement, flow control, etc. (Wang and Tronville, 2014). An atomizer was used to generate airborne Di(2-ethylhexyl) sebacate (DEHS) or Di(2-ethylhexyl) phthalate (DEHP) particles in the size range of 10–500 nm. DEHS could be used either as pure for producing larger particles, or diluted with isopropanol (IPA) for generating smaller particles down to 10 nm. DEHS droplets are spherical particles. In these experiments the DEHS concentration in IPA was 0.03 % for generating particles in the size range of 20 to 150 nm and 0.3% for 224 to 500 nm at laboratories A, B, C, D while 1% DEHS in IPA was used for the production of 10 to 150 nm and pure DEHS for particles above 150 nm was used at laboratory E. Indicative DEHS size distributions are presented in the supplementary material. A diffusion dryer was used to ensure evaporation of the solvent. Alternatively, a furnace was used to produce airborne silver particles in the size range of 3–30 nm. A 99.99% silver slug was inserted in a boat inside the furnace which was then heated to 800–1100°C; the silver evaporated and

then condensed into nanoparticles. Silver particles less than 30 nm are compact and close to spheres. Almost no difference on filtration efficiencies was measured for 30 nm silver particles and sintered silver spheres (Kim *et al.*, 2009). The particles were given the Boltzmann's equilibrium charging distribution by a krypton 85 or polonium 210 neutralizer.

Laboratories A, B, C, D performed the experiments with monodisperse particle flow as the challenging aerosol, whereas laboratory E determined the filter media filtration efficiency by challenging it with polydisperse particles. Indicative set up schematics are presented in Fig. 1. The monodisperse particles were obtained by classifying the aerosols from the generator using a differential mobility analyzer (DMA). DMA accuracy was verified with polystyrene latex beads and the results are presented in the supplementary material (Table S1). The monodisperse particles exiting the DMA mostly carried one electrical charge and were neutralized again by a neutralizer in order to minimize the filtration efficiency due to electrostatic forces. This approach reduced the electrostatic effect in filtration and the associated uncertainties. Specimens of the sheet filter medium were fixed in the test filter holder and subjected to the test air flow corresponding to the prescribed filtration face velocity. Particles were counted upstream and downstream from the filter using either two condensation particle counters (CPCs) in parallel, or using only one such counter to measure the upstream and downstream concentrations alternately.

Laboratory E determined the filtration efficiency using polydisperse particles. In this way, the particle distribution was measured from the upstream of downstream section of the filter. It should be mentioned that Laboratory E used a TSI Nanoscan 3910 operated in single mode for obtaining the data below 100 nm and the PMS LAS-X II, currently marketed as TSI 3340, for measuring the efficiency above 100 nm. The summary of the equipment used by the different laboratories is presented in Table 1.

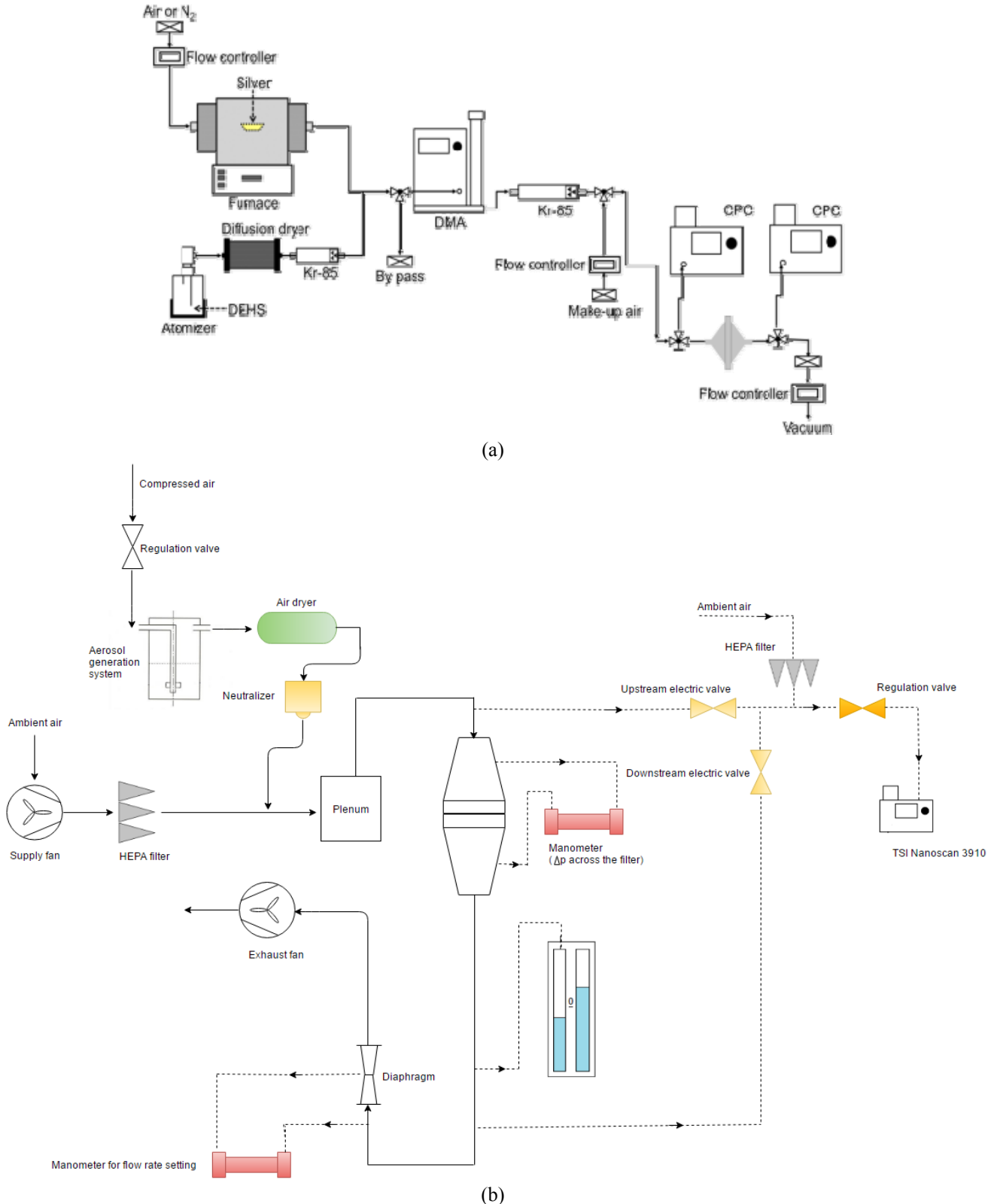
A pump positioned downstream drew the test aerosol through the test filter mounting assembly in both setups. Laboratory A, B, C and D used circular filter holders with diameter of 113 mm, while laboratory E used a squared one with the length of the side 300 mm. It should be noted that laboratory A used a 38 mm diameter filter holder for the tests performed at  $10 \text{ cm s}^{-1}$  to reduce the required flow rate.

Six different filter media were tested. A wire mesh was tested because it is homogeneous and it could be used as a reference filter. Two bag filter media were also tested; F7 made of PET (polyethylene terephthalate) which is a charged filter and F7 made of glass which is an uncharged one. Finally, three highly efficient pleatable non charged filter media were tested; F9 and H13 glass fiber filters and E11 PTFE (polytetrafluoroethylene) synthetic filter. The summary of the different filter properties is presented in Table 2.

When the filter is charged, the filtration due to the electrostatic forces could be substantial. The surface potential of the filter could be reduced down to zero by exposing it to IPA vapor (isopropanol) like Ohmi *et al.* (1994) and

Xiao *et al.* (2014) mentioned. The purpose of the neutralization is two folds. Firstly, the minimum filtration efficiency could be tested when the filter medium is neutralized, so that the reported efficiency is a conservative

value. Secondly, there is higher probability of variation if the filter samples are charged. Thus, the removal of the electrostatic charges from the medium improves the reliability of the test results.



**Fig. 1.** Test setup schematics (a) with monodisperse challenging aerosol & (b) with polydisperse challenging aerosol.

**Table 1.** Laboratories equipment.

Laboratory	Particle type	Particle Production	Particle Classification	Particle counting
A	DEHS	Home-made atomizer	TSI 3081 long DMA	TSI CPC model 3776 & 3775
	Silver	Carbolite Furnance	TSI 3080 nano DMA	
B	DEHS	TSI 3079 Atomizer	TSI 3081 long DMA	TSI CPC model 3776
	Silver	Home-made generator using furnace	TSI 3080 nano DMA	
C	DEHS	TSI model 3160 test rig internal type	TSI 3071 DMA	TSI CPC model 3772
	Silver	Self-built type with furnace and silver	TSI 3080 nano DMA	TSI CPC model 3775
D	DEHS	Compressed particle-free air feed through the Laskin nozzle	TSI 3082 DMA	TSI CPC model 3775
E	DEHS	TSI 3076 Constant output atomizer	TSI Nanoscan SMPS 3910 & PMS LAS-X II (TSI 3340)	

**Table 2.** Filters used in the inter-laboratory tests.

filter class	filter type		media type			glass fiber	PET synthetic
	bag filter	pleatable	synthetic				
			non-charged	charged	discharged/non charged		
Mesh			X				
F7 PET	X			X			X
F7 glass	X		X			X	
F9		X	X			X	
E11		X	X				X
H13		X	X			X	

## ANALYSES

### Filtration Efficiency Calculation

The line losses for the upstream and downstream sampling might be different. The difference can be significant due to the diffusion loss when the particle size is very small. In addition, some particles might be deposited at the inlet, outlet or walls of the filter holder. The upstream and downstream CPCs may have different counting efficiencies. Therefore, it was important to establish correlation ratios by performing the measurement without any filter medium in the filter holder. Then the filtration efficiency was measured with the filter placed inside the holder and calculated according to Wang and Tronville (2014).

### Statistical Analysis of the Experimental Results

The reliability of the measurement method was verified by applying the statistical analysis according to ISO 5725-2. More details could be retrieved from the ISO 5725-2.

Overall, every test result for each particle size ( $Y$ ) was assumed to be the sum of the general mean filtration efficiency ( $m$ ), the laboratory component of bias under repeatability conditions ( $b$ ) and the random error under repeatability conditions ( $e$ ). In order to define the precision of the test procedure the estimates of the repeatability ( $s_r$ ) and reproducibility ( $s_R$ ) deviations were determined. The equations are presented below:

$$Y = m + b + e, \quad (1)$$

$$s_L = \sqrt{\text{Var}(b)}, \quad (2)$$

$$s_r = \sqrt{\text{Var}(e)}, \quad (3)$$

$$s_R = \sqrt{(s_L^2 + s_r^2)}, \quad (4)$$

where  $s_L$  is the deviation between laboratories.

Before determining the aforementioned deviations, the outliers and stragglers were defined, using Mandel's  $h$  &  $k$  (Mandel, 1985), Grubb's (Grubbs, 1950; Grubbs and Beck, 1972) and Cochran's analyses (Cochran, 1941). Wilrich (2011) provided the detailed procedure to apply these tests. The outliers were excluded from the final calculation of the standard deviations. Mandel's  $h$  &  $k$  is a graphical consistency technique. It describes the variability of the results from the measurement method and helps the laboratory evaluation. In this analysis  $h$  is the between-laboratory consistency statistic while  $k$  is the within laboratory consistency statistic. The other tests are numerical consistency techniques. In Cochran's test it is assumed that only small differences exist in the within laboratory variances. Grub's test determines whether the largest or smallest observations are outliers or stragglers. The values calculated from the techniques above were compared with the critical values at the significant level of 0.05 to define the stragglers and 0.01 for outliers. A low number of outliers and small deviation values indicate that the test method is reliable.

### Anova Analysis

Analysis of variance (Anova) is a method to test the null hypothesis that the means of several groups are equal (Kutner et al., 2004). F test is used to test this hypothesis.

If the calculated F value is greater than the critical F value at the significant level of 0.05, the null hypothesis is rejected and at least one group has a different mean than at least another.

### Regression Analysis

Regression analysis is a statistical method to establish the relationship among variables. If the effect of several variables on another is tested, it is called a multiple regression. The hypothesis that the dependent variable Y has the following relationship (5) with the independent variables x is made (Seber et al., 2003)

$$Y = a + a_1x_1 + a_2x_2 + \dots + a_nx_n + e_1, \quad (5)$$

where  $a$  is the constant coefficient,  $a_n$  are the coefficients of the variables and  $e_1$  is the error term.

Regression analysis provides estimates for the values of  $a$  and  $a_n$  coefficients. The independent variable affects the dependent variable, if the calculated P value of the parameter is less than the critical P value at 0.05 significant level. The relationship is defined by their coefficient.

### Correlation Analysis

The correlation analysis reveals the correlation coefficient which evaluates how strong the relation between two variables is. It measures the degree to which two variables move in relation to each other. The correlation coefficient values are from  $-1$  to  $1$ . The closer to one it is, the stronger the correlation between the two variables is (Microsoft Excel tutorial, 2010). The sign indicates the type of the correlation.

### Determination of the Figure of Merit

Hinds (1998), Dhaniyala and Liu (1999) and Wang et al. (2008) evaluated the filtration performance by obtaining the figure of merit. The numerator is a measure of the filtration efficiency, thus the higher the figure of merit the better the performance of the filter. The figure of merit could be calculated as:

$$\gamma = -\ln(P/100)/\Delta P \quad (6)$$

where  $P$  is the penetration of the media at defined particle size in % and  $\Delta P$  is the pressure drop of the media in Pa.

## RESULTS

### Round Robin Test Results Analysis

#### Filtration Efficiency Analysis

Five different laboratories tested the filtration efficiencies of different filter media at  $2 \text{ cm s}^{-1}$ ,  $5 \text{ cm s}^{-1}$  and  $10 \text{ cm s}^{-1}$  face velocity. Laboratories D and E did not participate in the tests using silver aerosols but E provided efficiency data down to  $10 \text{ nm}$  using DEHS diluted with IPA. Laboratory E used two different measurement principles to size the particles, thus, on the same efficiency curve for E both mobility diameter and optical diameter are presented, although no noticeable gap was observed between the sections corresponding to the two different instruments. Indicative results are presented in Fig. 2. The rest of the results are presented in the supplementary material. The theoretical filtration efficiency is presented with the thick solid line and the model equations are presented by Sachinidou et al. (2017). Diffusion was the main mechanism for the filtration efficiency below  $100 \text{ nm}$ . The smaller the particle was, the higher mobility it acquired, therefore, the filtration efficiency was high for small particles. In this size range, the efficiency dropped with the increasing face velocity due to the shorter time that the particle could diffuse. From  $100 \text{ nm}$  to  $224 \text{ nm}$  the main filtration mechanism was the interception and above this range impaction started to increase. Thus, the most penetrating particle size was around  $224 \text{ nm}$ . The experimental results from laboratories A, B and C had low variances. The results from Laboratory D showed high within laboratory deviation, regardless the particle size or face velocity, which could be attributed to a problem in their setup affecting the repeatability. Laboratory E often measured lower filtration efficiency compared to other laboratories.

In order to verify that the test procedure was reliable the

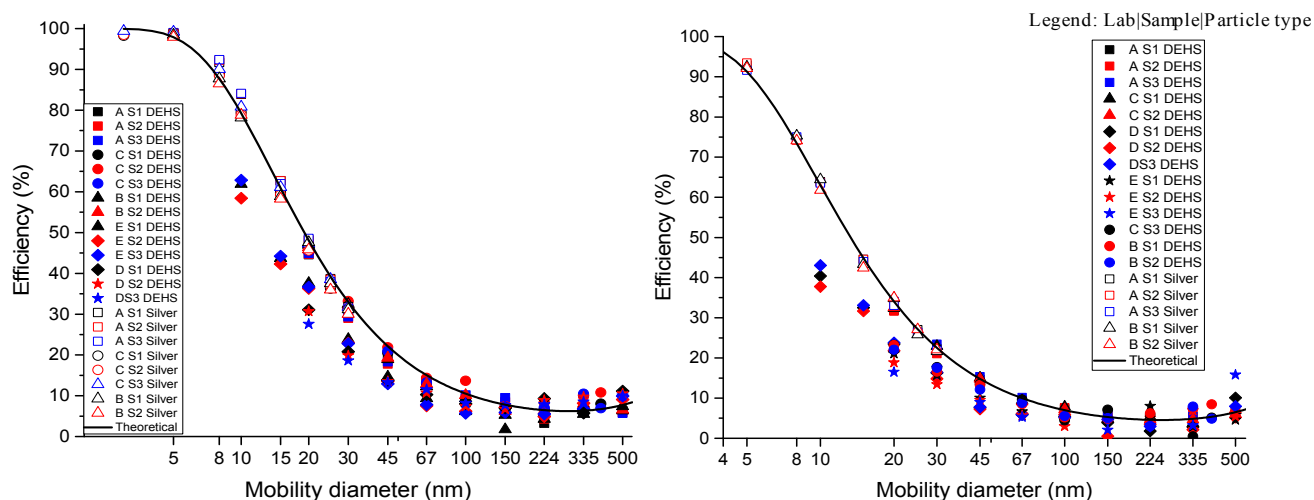


Fig. 2. Filtration efficiency test results for the wire mesh at  $5 \text{ cm s}^{-1}$  (left) and  $10 \text{ cm s}^{-1}$  (right).

experimental results were analyzed according to the section Statistical analysis of the experimental results.

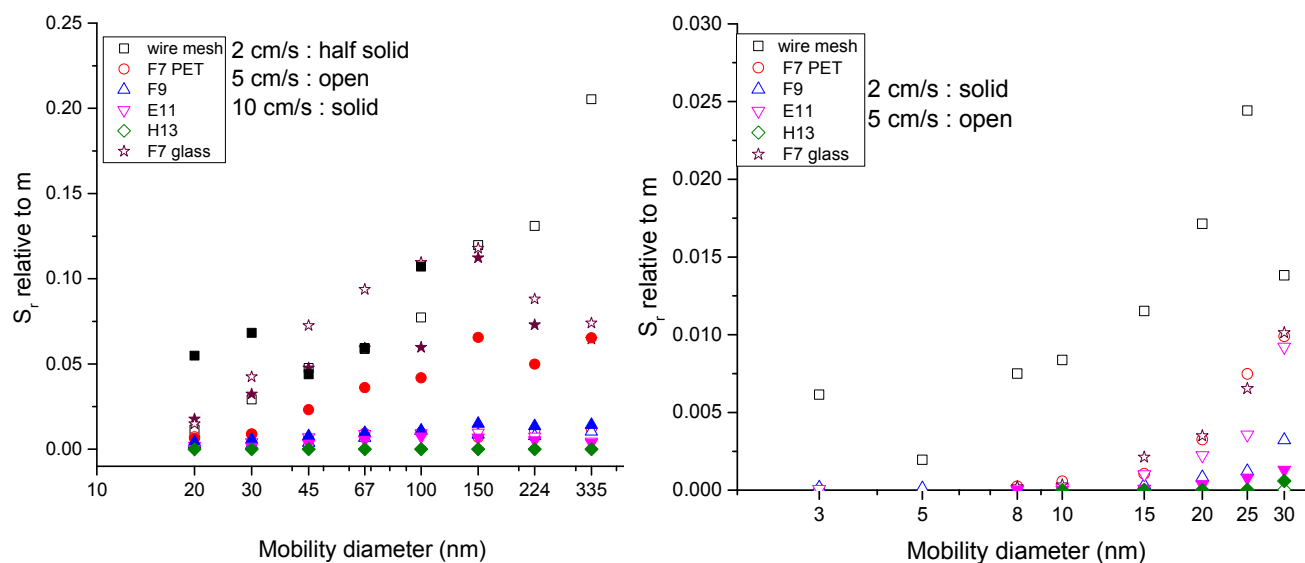
To acquire statistically reliable results, outliers and stragglers were defined for the tests using DEHS particles, for which all the five laboratories provided data therefore the sample number is five. The general mean filtration efficiency and the repeatability and reproducibility deviations, as fractions of the general mean filtration efficiency, were calculated and the results are presented in Table 3, Figs. 3 and 4 respectively. Increase of the face velocity slightly deteriorated the results leading to higher deviations. However, deviations strongly depended on the particle size. Filtration efficiency is a function of the particle size, thus close to the most penetrating particle size (MPPS) the filtration efficiency is low and the experimental errors could

significantly affect the measurement. Therefore, in this size range the deviations were higher.

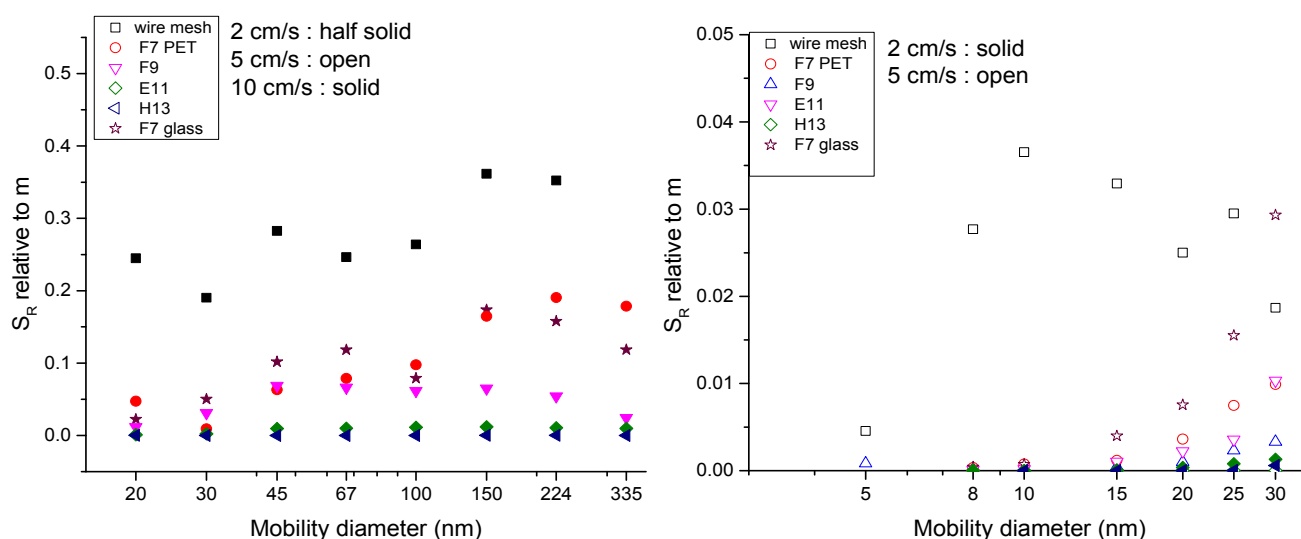
The repeatability deviation is less than 0.1 and 0.03 of the absolute magnitude of the filtration efficiency for almost all the filters and particles sizes for DEHS and silver challenging particles respectively. When silver particles challenged the filter, the reproducibility deviation is below 0.05 for all the cases. For DEHS, the experimental results showed less than 0.2 deviation compared to the absolute magnitude of the filtration efficiency regardless the particle size or face velocity for all the filters apart from the wire mesh. Despite the fact that the absolute magnitude of reproducibility deviation for the wire mesh was low (below 2.5% in most of the cases), the relative deviation was close to 0.2 at 5 cm s<sup>-1</sup> and 0.3 at 10 cm s<sup>-1</sup> because the filtration

**Table 3.** Average filtration efficiency for every tested filter media in the size range of 20–500 nm.

Filters	wire mesh		F7 PET		F7 glass		F9		E11		H13	
Velocity (cm s <sup>-1</sup> )	5	10	5	10	5	10	5	10	2	5	2	5
Particle size (nm)	Filtration efficiency (%) - DEHS											
20	43.2	25.5	96.9	91.5	97.2	94.6	97.8	94.6	99.8	98.6	100.0	100.0
30	27.0	18.4	92.4	86.5	92.1	87.2	94.6	87.2	99.3	96.4	100.0	100.0
45	17.0	11.7	88.7	77.1	83.2	76.7	88.3	76.7	97.5	93.2	100.0	100.0
67	11.7	7.9	84.2	73.3	73.5	67.6	80.2	67.6	95.6	90.9	100.0	100.0
100	8.1	5.7	83.8	71.6	63.8	62.0	71.0	62.0	94.4	90.3	100.0	99.9
150	7.2	4.7	85.4	68.4	57.0	58.1	66.7	58.1	94.2	91.4	100.0	99.9
224	6.3	4.5	86.0	66.9	54.1	61.3	67.5	61.3	95.6	94.6	100.0	100.0
335	7.6	4.2	88.0	69.5	57.7	69.5	73.4	69.5	97.4	97.2	100.0	100.0
Particle size (nm)	Filtration efficiency (%) - Silver											
3	98.7	N/A	100.0	N/A	N/A	N/A	100.0	N/A	N/A	100.0	N/A	N/A
5	98.8	N/A	N/A	N/A	N/A	N/A	100.0	N/A	N/A	N/A	N/A	N/A
8	89.8	N/A	100.0	N/A	100.0	N/A	100.0	N/A	100.0	100.0	N/A	N/A
10	81.0	N/A	99.9	N/A	99.9	N/A	99.9	N/A	100.0	99.9	100.0	100.0
15	60.6	N/A	99.5	N/A	99.4	N/A	99.6	N/A	100.0	99.6	100.0	100.0
20	47.3	N/A	98.3	N/A	98.7	N/A	98.8	N/A	99.9	98.8	100.0	100.0
25	37.6	N/A	96.8	N/A	97.3	N/A	97.6	N/A	99.7	97.9	100.0	100.0
30	31.3	N/A	95.1	N/A	95.7	N/A	96.1	N/A	99.4	96.5	100.0	100.0



**Fig. 3.** Repeatability deviation as a fraction of the average efficiency (DEHS in the left panel & silver in the right panel).



**Fig. 4.** Reproducibility deviation as a fraction of the average efficiency (DEHS in the left panel & silver in the right panel).

efficiency was low in the size range around the MPPS, thus, the experimental errors could impose a stronger effect in the measurement.

Overall, there were limited outliers (see supplementary material) for all the filters and in the whole particle size range regardless the challenging particle material. The repeatability deviation was mostly less than 0.1 of the absolute efficiency regardless the particle size, face velocity or challenging particle material. The only exception is for the wire mesh because filtration efficiency is low, thus, the error can highly contribute to the deviation. The reproducibility deviation was more significant. Deviations for the silver particles were smaller compared to the DEHS because only the three laboratories with the same setup participated. Also, the filtration efficiency is higher for smaller particles, thus, experimental errors are not so important compared to the absolute magnitude of the efficiency. Possible contributors of these deviations included the inhomogeneity of the filter media samples, different instruments and experimental setups in the participating laboratories, inherent instrument uncertainties, etc.

Since laboratory E used a different experimental setup and laboratory D's data showed high variability, the data measured by laboratories A, B and C were used to calculate the repeatability and reproducibility deviations and the results are presented in Fig. 5. Relative repeatability and reproducibility deviations were below 0.05 and 0.1 respectively, for all the different filters apart from the wire mesh because the filtration efficiency was very low and experimental errors greatly influenced the results. Overall, lower relative deviations were calculated using the data set of the three laboratories which utilized the same particle measurement systems and filter holders. Discussions of more parameters are presented in the sensitivity analysis.

#### Pressure Drop Analysis

The pressure drop measured by the different laboratories is presented in Table 4. The wire mesh is a highly homogeneous filter, thus, the deviations among each

laboratory measurement were low. Variation among the data for the other filters was observed. Laboratory A measured higher pressure drop compared to the other laboratories in many cases which could possibly be attributed to the measuring range of the instrument. The rest of the laboratories measured close pressure drop in almost all the cases. Variation among the results for the same filter could be partially explained by the filter inhomogeneity.

#### Figure of Merit of the Tested Filters

Inter-laboratory test results of the filtration efficiency and pressure drop were presented in the last two sections. The variations in the filtration efficiency and pressure drop may be attributed to the inhomogeneity of the filter samples. Inhomogeneity influences less the figure of merit than the efficiency or pressure drop, because if the inhomogeneity causes higher pressure drop for a specific piece of sample, then the corresponding efficiency should also be higher.

The averaged values of figure of merit (three different tested samples per filter) at different velocities and for the particle sizes of 45, 100, and 150 nm are presented in Table 5. The comparison can be performed for the same filter media at the same velocity and particle size. According to the results there was not a specific pattern indicating that a single laboratory always obtained a value far from the others. The deviation among the laboratories was lower than 10% in the majority of the tested cases. However, there were cases such as F7 glass or E11 where the range of the figure of merit was big. Therefore, there was not a clear indication that the higher deviations among the experimental results were exclusively due to filter inhomogeneity.

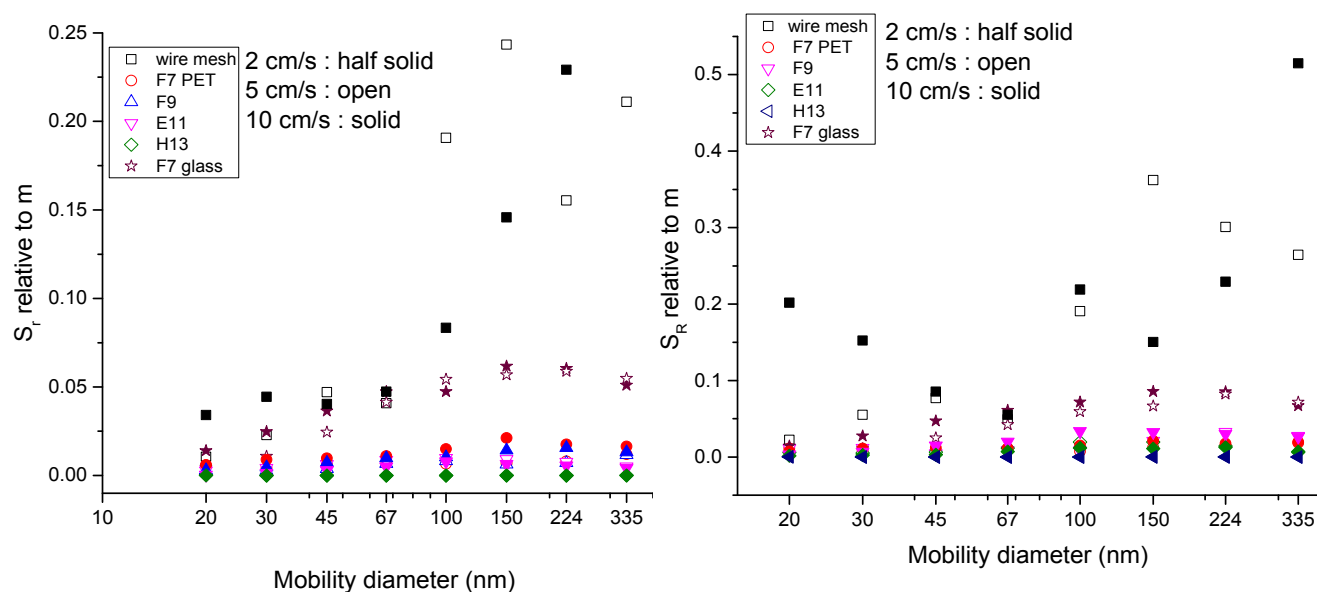
#### Sensitivity Analysis

##### Particle Density Effect on Filtration Efficiency

The particle density plays a role in determining the inertia of the particles and therefore the impaction and gravitational settling mechanisms. The effects are not substantial for nanoparticles.

Laboratories A, B and C carried out experiments for





**Fig. 5.** Repeatability (left panel) and reproducibility (right panel) deviations as fractions of the average efficiency using the data of the three laboratories A, B, C and DEHS as challenging particles.

**Table 4.** Average filter media pressure drop measured by the different laboratories.

	Pressure drop (Pa)					
	Velocity ( $\text{cm s}^{-1}$ )	A	B	E	D	C
wire mesh	5	57	54	55	51	58
	10	112	108	112	102	112
F7 PET	5	36	22	21	25	23
	10	53	46	37	46	N/A
F7 glass	5	32	26	21	21	26
	10	59	50	42	44	52
F9	5	55	41	45	41	44
	10	97	85	83	79	88
E11	2	34	24	35	23	25
	5	73	57	78	57	59
H13	2	135	120	129	110	116
	5	334	294	292	266	284

both DEHS ( $0.9 \text{ g cm}^{-3}$ ) and silver particles ( $10.49 \text{ g cm}^{-3}$ ) in the size range of 20–30 nm and the filtration efficiencies were compared to prove the aforementioned argument. The results showed that the DEHS and silver particles of the same electrical mobility size had almost the same filtration efficiencies, with the differences below 3%, thus the particle density did not notably affect the filtration efficiency in the size range well below 100 nm.

#### *The Effect of Temperature and Relative Humidity on Filtration Efficiency*

The temperature affects the diffusion coefficient of the particles, therefore the filtration efficiency. The variation was not expected to be wide since the round robin tests were at room conditions, therefore the effect was expected to be limited. In addition, the relative humidity affects the air density, thus, it might also affect the filtration efficiency.

All the partner laboratories collected the relative humidity and temperature information during the round robin tests which allowed the comparison of the results measured at different relative humidity and temperature levels.

The range of temperature was about 20–30°C and 7–50% for relative humidity. Laboratory D and E measured the temperature and relative humidity for each tested filter and at each particle size. Their results were analyzed with multiple regression, using the built-in function in Excel, so as to quantify the relationship among the parameters and the filtration efficiency. If the calculated p-value of a parameter was less than 0.05 (significant level), this parameter affected the filtration efficiency significantly. Otherwise, there was no clear indication that this parameter was linked to the filtration efficiency. The results from the regression showed that there was no definitive indication that temperature and relative humidity affected the filtration efficiency in these ranges, as Kim *et al.* (2006) and Yang and Lee (2004) concluded. The regression for each filter is presented in the supplementary material.

#### *Effect of the Concentration of the Challenging Aerosol on Filtration Efficiency*

Concentration may affect the filtration efficiency depending on the range. If the challenging aerosol concentration was too high, there could be particle agglomeration or loading effect on the filter which might affect the filtration efficiency like Kim *et al.* (2009) and Buha *et al.* (2013) stated. If in the proper range, the challenging aerosol concentration should not affect the filtration efficiency. It should be mentioned that high concentration could overload the particle sizer or increase the measurement errors from the particle counter due to the switch to a photometric measurement mode.

Data from the partners during the inter-laboratory tests were analyzed to test if the challenging concentration



**Table 5.** Averaged figure of merit at different velocities.

		5 cm s <sup>-1</sup>					10 cm s <sup>-1</sup>					2 cm s <sup>-1</sup>	
		45 nm											
	wire mesh	F9	F7 PET	F7 glass	E11	H13	wire mesh	F9	F7 glass	E11	H13		
A	0.4	3.9	6.2	6.6	3.8	2.9	0.14	1.7	2.7	11.8	9.5		
E	0.3	3.4	8.4	6.6	3.1	2.6	0.07	1.4	2.6	9.3	7.5		
C	0.3	4.8	9.9	8.9	4.8	3.4	0.12	1.9	3.4	15.9	10.9		
D	0.4	5.4	8.5	7.7	4.7	3.9	0.10	1.7	2.6	16.5	11.6		
B	0.4	5.2	9.5	8.1	4.9	3.1	0.15	1.8	3.0	16.0	10.8		
		100 nm											
	wire mesh	F9	F7 PET	F7 glass	E11	H13	wire mesh	F9	F7 glass	E11	H13		
A	0.2	2.3	5.3	3.6	3.1	2.2	0.07	1.1	1.6	9.0	7.2		
E	0.1	2.6	8.1	4.7	3.3	2.5	0.05	1.2	1.9	8.3	6.3		
C	0.2	2.9	8.3	4.7	4.3	2.6	0.05	1.1	1.9	12.6	8.3		
D	0.2	3.0	6.6	3.9	3.8	2.8	0.04	1.0	1.3	11.9	8.6		
B	0.2	3.1	8.2	4.1	3.9	2.4	0.07	1.1	1.6	11.5	8.0		
		150 nm											
	wire mesh	F9	F7 PET	F7 glass	E11	H13	wire mesh	F9	F7 glass	E11	H13		
A	0.2	2.1	5.6	2.9	3.3	2.1	0.05	1.0	1.4	8.6	6.7		
E	0.1	2.2	8.7	3.8	3.2	2.1	0.03	1.0	1.5	8.2	6.5		
C	0.1	2.7	8.3	4.0	4.6	2.6	0.05	1.1	1.7	12.8	8.0		
D	0.1	2.6	6.2	3.3	4.1	2.7	0.04	1.0	1.1	11.6	8.1		
B	0.1	2.7	8.9	3.4	4.1	2.5	0.05	1.0	1.4	11.6	7.5		

correlated with the filtration efficiency. The correlation coefficient was calculated using the correlation function in Excel. According to the results presented in the supplementary material, the absolute value of the correlation coefficient was in almost all the cases smaller than 0.5, thus, there was no clear indication that the challenging particle concentration affected the filtration efficiency in the range of tens to tens of millions particles per cc.

#### *Challenging Particle Size Distribution, SAFR and Neutralization Efficiency*

When the DMA is used as a classifier, its set parameters should be studied to ensure the minimization of the measurement artifacts. Sheath to aerosol flow ratio (SAFR) could affect the monodispersity of the challenging particle flow based on the generated particle size distribution. According to Sachinidou *et al.* (2017) a SAFR above or equal to five could minimize the artifacts due to this factor for the investigated filtration tests and the particle size distribution at the DMA inlet would have marginal effect on the measurement. In addition, neutralization efficiency for the particles entering the DMA was crucial so as to avoid bigger multiply charged particles to enter the challenging particle flow.

All the laboratories maintained a SAFR above or equal to five and qualified that the neutralizer functioned properly (the qualification data are presented in the supplementary material), thus the aforementioned parameters were not expected to contribute significantly to the deviation among the experimental results from the different laboratories.

#### *Flow Distribution*

Flow distribution depends on the filter holder geometry,

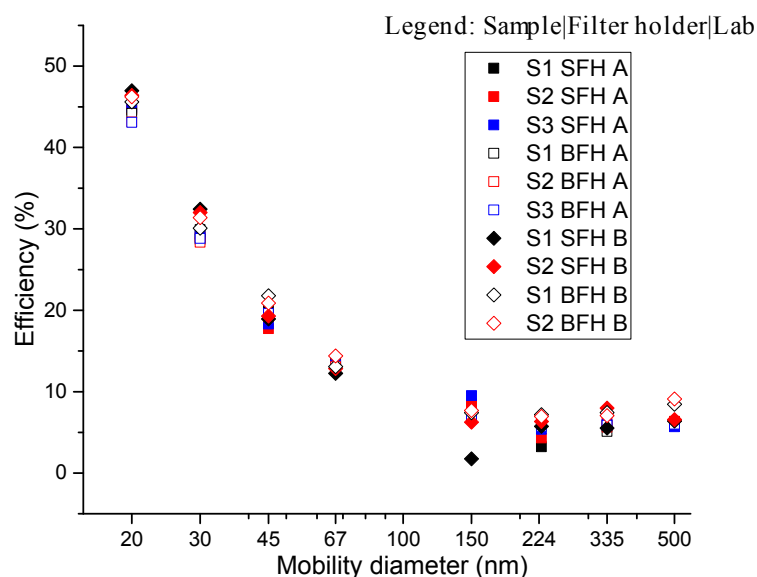
thus the filter holder size and shape could possibly affect the flow velocity distribution.

The test filter media with different surface areas may possess different uniformity levels thus leading to variation in the test results. Laboratories A and B tested the wire mesh filter media, which is a highly homogeneous filter, at 5 cm s<sup>-1</sup> using two different filter holders (D<sub>min</sub> = 38 mm and D<sub>max</sub> = 113 mm). The results presented in Fig. 6 showed a good agreement among the filtration efficiencies measured with different filter holders in the whole particle size range for the wire mesh, thus, indicating no obvious link between the filter surface area and filtration efficiency if the filter medium was highly uniform. Similar results were presented by Sachinidou *et al.* (2017) supporting that the face velocity distribution was homogeneous upstream the filter media.

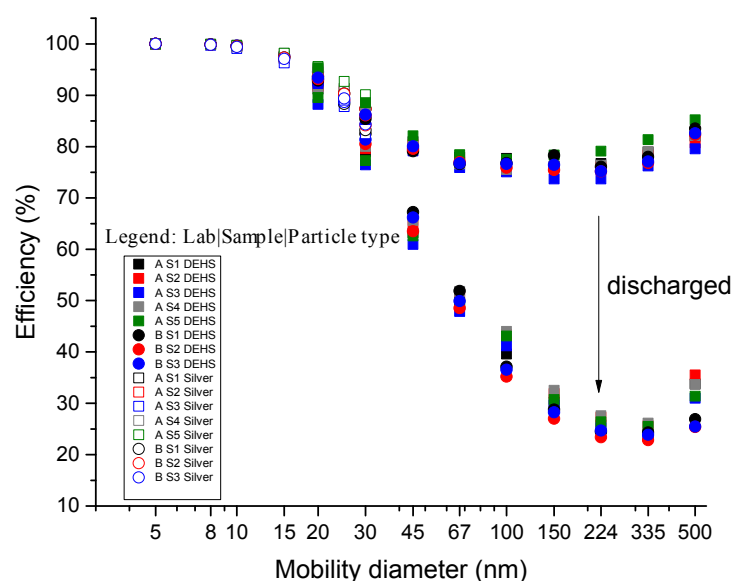
A regression analysis was performed on the round robin results to evaluate if the filter holder shape and area affected the filtration efficiency. This analysis revealed a possible link; however, it is difficult to conclude that the filter holder geometry was the crucial parameter since the test setups were different.

#### *Charge on the Filter*

The charge on the filter could increase the filtration efficiency due to the electrostatic filtration mechanism. Therefore, it was crucial to reduce the surface potential of the filter down to zero. The challenging particles acquired the Boltzmann's equilibrium distribution, thus, the average charge of the particles was zero. Thus, the filtration efficiency decreased if the filter was discharged as Brown (1993) stated. The reduction in the filtration efficiency is presented in Fig. 7 and it is crucial for particles above 30 nm.



**Fig. 6.** Filtration efficiency of the wire mesh at  $5 \text{ cm s}^{-1}$  measured in filter holders with different areas (SFH: Small Filter Holder, BFH: Big Filter Holder).



**Fig. 7.** F7 filtration efficiency at  $10 \text{ cm s}^{-1}$  for charged and discharged filter.

## CONCLUSIONS

Upon the completion of the round robin tests, the experimental results were analyzed so as to determine the reliability of the test method. Statistical analysis revealed a few stragglers or outliers in most of the cases. Laboratory D showed high within-laboratory deviation and E measured lower filtration efficiency in most of the cases. Furthermore, reproducibility and repeatability deviations were below 0.2 and 0.05 compared to the mean filtration efficiency.

A further sensitivity analysis was performed to investigate the possible reasons that could contribute to the variation among the experimental results. There was no clear indication that relative humidity, temperature or upstream challenging particle concentration affected the

filtration efficiency in the range of the parameters observed in the tests. Particle density did not affect the filtration efficiency notably in the nanometer particle size range. The filter charge status exhibited a crucial effect on the filtration efficiency, thus, the filter should be discharged to exclude the electrostatic filtration mechanism and to measure the worst-case filtration efficiency.

The face velocity profile for a circular shape filter holder was simulated in Fluent by Sachinidou *et al.* (2017) and the results exhibited no obvious effect by the velocity uniformity on the filtration efficiency for the two studied media which were highly uniform. This was supported as well by the experiments which showed marginal deviation between the results obtained with the filter holders of two different sizes. However, regression analysis revealed that

the filtration efficiency may be affected by the relation of the flow velocity distribution difference between the squared and circular filter holders. However, this was not conclusive since different setups were used.

There were a few cases such as E11 or F7 PET where the range of figure of merit was large. Therefore, there was not a clear indication that the higher deviations among the experimental results are exclusively due to filter inhomogeneity.

Last but not least, the differences between the measurement approach used by laboratories A, B, C, D and that used by E, which were the monodisperse vs polydisperse flow and CPC vs. NanoScan SMPS & optical particle spectrometer, could possibly explain partially the deviations among the experimental results. Thus, repeatability and reproducibility deviations were calculated with the subsets data of the three laboratories A, B and C to validate the aforementioned argument. The comparison showed that when experimental setups with the same particle generation, classifying and counting systems and comparable filter holders were used, the filtration tests led to low uncertainties. If setups with different particle measurement systems and filter holders were used, the filtration tests led to generally consistent efficiency curves but the uncertainty might be higher.

#### ACKNOWLEDGEMENT

The study is supported by the European Commission in the frame of mandate M/461 “Standardization activities regarding nanotechnologies and nanomaterials.”

#### SUPPLEMENTARY MATERIAL

Supplementary data associated with this article can be found in the online version at <http://www.aaqr.org>.

#### REFERENCES

- American Society of Heating, Refrigerating and Air-Conditioning Engineers (2017) ANSI/ASHRAE Standard 52.2-2017 “Method of Testing General Ventilation Air Cleaning Devices for Removal Efficiency by Particle Size”, ASHRAE, Atlanta.
- Brown, R.C. (1993). *Air filtration: An integrated approach to the theory and applications of fibrous filters*. Pergamon Press, Oxford, New York.
- Buha, J., Fissan, H. and Wang, J. (2013). Filtration behavior of silver nanoparticle agglomerates and effects of the agglomerate model in data analysis. *J. Nanopart. Res.* 15: 1709.
- Cochran, W.G. (1941). The distribution of the largest of a set of estimated variances as a fraction of their total. *Ann. Eugen.* 11: 47–52.
- Dhaniyala, S. and Liu, B.Y.H. (1999). Investigations of particle penetration in fibrous filters Part II. Theoretical. *J. IEST* 42: 40–46.
- European Committee for Standardization (2009). EN 1822-3:2009 “High efficiency air filters (EPA, HEPA and ULPA) - Part 3: Testing flat sheet filter media”. CEN, Brussels.
- European Committee for Standardization (2012). EN 779:2012 “Particulate air filters for general ventilation—determination of the filtration performance”. CEN, Brussels.
- Grubbs, F.E. (1950). Sample criteria for testing outlying observations. *Ann. Math. Statist.* 21: 27–58.
- Grubbs, F.E. and Beck, G. (1972). Extension of sample sizes and percentage points for significance tests of outlying observations. *Technometrics* 14: 847–854.
- Hinds, W.C. (1998). *Aerosol Technology*, 2nd edition. Wiley-Interscience, New York.
- Huang, S.H., Chen, C.W., Chang, C.P., Lai, C.Y. and Chen, C.C. (2007). Penetration of 4.5 nm to 10  $\mu\text{m}$  aerosol particles through fibrous filters. *J. Aerosol Sci.* 38: 719–727.
- International Organization for Standardization (1994). ISO 5725-2:1994. Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for determination of repeatability and reproducibility of a standard measurement method.
- International Organization for Standardization (2016). ISO 16890-2:2016 “Air filters for general ventilation -- Part 2: Measurement of fractional efficiency and air flow resistance”. ISO, Geneva.
- International Organization for Standardization (2011). ISO 29463-5:2011 “High-efficiency filters and filter media for removing particles in air—Part 5: test method for filter elements”. ISO, Geneva.
- International Organization for Standardization (2013). ISO 29461-1:2013 “Air intake filter systems for rotary machinery—test methods—Part 1: static filter elements”. ISO, Geneva.
- Kim, C.S., Bao, L., Okuyama, K., Shimada, M. and Niinuma, H. (2006). Filtration efficiency of a fibrous filter for nanoparticles. *J. Nanopart. Res.* 8: 215–221.
- Kim, S.C., Wang, J., Emery, M.S., Shin, W.G., Mulholland, G.W. and Pui, D.Y.H. (2009). Structural property effect of nanoparticle agglomerate on particle penetration through fibrous filter. *Aerosol Sci. Technol.* 43: 344–355.
- Kutner, M., Nachtsheim, C., Neter, J. and Li, W. (2004). *Applied Linear Statistical Models*, McGraw-Hill/Irwin, Homewood, IL.
- Lore, M.B., Sambol, A.R., Japuntich, D.A., Franklin, L.M. and Hinrichs, S.H. (2011). Inter-laboratory performance between two nanoparticle air filtration systems using scanning mobility particle analyzers. *J. Nanopart. Res.* 13: 1581–1591.
- Mandel, J. (1985). A new analysis of interlaboratory test results. ASQC Quality Congress Transaction—Baltimore. pp. 360–366.
- Maze, B., Tafreshi, H. V., Wang, Q. and Pourdeyhimi, B. (2007). A simulation of unsteady-state filtration via nanofiber media at reduced operating pressures. *J. Aerosol Sci.* 38:550-571.
- Microsoft Excel tutorial (2010).
- Ohmi, T., Sudoh, S. and Mishima, H. (1994). Static charge removal with IPA solution. *IEEE Trans. Semicond.*

- Manuf.* 7: 440–446.
- Thomas, D., Mouret, G., Cadavid-Rodriguez, M.C., Chazelet, S. and Bemer, D. (2013). An improved model for the penetration of charged and neutral aerosols in the 4 to 80 nm range through stainless steel and dielectric meshes. *J. Aerosol Sci.* 57: 32–44.
- Sachinidou, P., Bank, Y.K. and Wang, J. (2017). An integrative model for the filtration efficiencies in realistic tests with consideration of the filtration velocity profile and challenging particle size distribution. *Aerosol Sci. Technol.* 51: 178–187.
- Seber, G.A.F. and Lee, A.J. (2003). *Linear Regression Analysis*. John Wiley and Sons. New Jersey
- Steffens, J. and Coury, J.R. (2007a). Collection efficiency of fiber filters operating on the removal of nano-sized aerosol particles: I—homogeneous fibers. *Sep. Purif. Technol.* 58: 99–105.
- Steffens, J. and Coury, J.R. (2007b). Collection efficiency of fiber filters operating on the removal of nano-sized aerosol particles II. Heterogeneous fibers. *Sep. Purif. Technol.* 58: 106–112.
- Wang, J., Chen, D.R. and Pui, D.Y.H. (2007). Modeling of filtration efficiency of nanoparticles in standard filter media. *J. Nanopart. Res.* 9: 109–115.
- Wang, J., Kim, S.C. and Pui, D.Y.H. (2008). Investigation of the figure of merit for filters with a single nanofiber layer on a substrate. *J. Aerosol Sci.* 39: 323–334.
- Wang, J. and Tronville, P. (2014). Toward standardized test methods to determine the effectiveness of filtration media against airborne nanoparticles. *J. Nanopart. Res.* 16: 2417.
- Wilrich, P. (2011). Critical values of Mandel's  $h$  and  $k$ , the Grubbs and the Cochran test statistic. *AStA Adv. Statist. Anal.* 97: 1–10.
- Xiao, H., Song, Y. and Chen, G. (2014). Correlation between charge decay and solvent effect for melt-blown polypropylene electret filter fabrics. *J. Electrostat.* 72: 311–314.
- Yang, S. and Lee, G.W.M. (2004). Filtration characteristics of a fibrous filter pretreated with anionic surfactants for monodisperse solid aerosols. *J. Aerosol Sci.* 36: 419–437.

Received for review, March 10, 2017

Revised, June 1, 2017

Accepted, June 2, 2017