

# 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>Analytical Chemistry Laboratory, Empa, Dübendorf, 8600, Switzerland, <sup>2</sup>Institute of Environmental Analytical Chemistry Laboratory, ETH Zurich, Zurich, 8093, Switzerland, <sup>3</sup>Particle Technology Laboratory, Department of Mechanical Engineering, University of Minnesota, Minneapolis, MN 55414, USA, <sup>4</sup>Politecnico di Torino DENERG, Corso Duca degli Abruzzi, 24 10129 Turin, Italy, <sup>5</sup>Unifil AG, Filtertechnik, Industriestrasse 1, 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 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. Results statistical analysis 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. 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.

**Key words:** Filtration efficiency, Interlaboratory tests, Sensitivity analysis, Statistical analysis

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\* Corresponding author. Tel: 1+41 44 633 36 21  
E-mail address: jing.wang@ifu.baug.ethz.ch

## 39 1 Introduction

40  
41 Filtration of airborne nanoparticles is crucial due to the increased produced quantities  
42 from material synthesis and combustion emissions (Wang and Tronville (2014)). More small par-  
43 ticles are being produced compared to the past due to the blossom of the field of nanotechnology.  
44 Many experimental and theoretical studies for particles down to single digit nanometers have al-  
45 ready been performed by many researchers such as Kim et al. (2009), Wang et al. (2007), Thom-  
46 as et al. (2013) Huang et al. (2007), Steffens and Coury (2007)<sup>a,b</sup>.

47 Filtration testing is very challenging because many parameters can affect the filtration ef-  
48 ficiency. Sachinidou et al. (2017) concluded that particle size distribution and charge could pos-  
49 sibly affect the filtration test accuracy. Kim et al. (2006) and Yang and Lee (2004) showed that  
50 relative humidity did not influence the filtration efficiency. However, the charge status of the fil-  
51 ter media could affect the filtration efficiency as stated by Brown (1993), Lore et al. (2011),  
52 Huang et al. (2007), Maze et al. (2007) showed that flow temperature can alter filtration efficien-  
53 cy. Thus, it is crucial to determine a reliable procedure for the filtration test which could mini-  
54 mize the artifacts. Even though there are a number of standards for testing air filters that cover a  
55 large particle size range like ASHRAE 52.2-2017, EN 1822:2009, EN 779:2012, ISO 16890:2016  
56 ISO 29461-1:2013, ISO 29463-3:2011 up to several micrometers, there is no standard focusing  
57 on the filtration of nanoparticles down to single digit nanometers. It is very challenging to devel-  
58 op such a procedure.

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

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

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

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## 79 2 Filtration efficiency tests

80  
81 The test system for filtration efficiency of airborne particles usually involves an aerosol  
82 generation part, particle measurement part, filter holder system, and other parts for pressure  
83 measurement, flow control, etc. (Wang and Tronville, 2014). An atomizer was used to generate  
84 airborne Di(2-ethylhexyl) sebacate (DEHS) or Di(2-ethylhexyl) phthalate (DEHP) particles in the  
85 size range of 10-500 nm. DEHS could be used either as pure for producing large particles, or di-  
86 luted with isopropanol (IPA) for generating smaller particles down to 10 nm. DEHS droplets are  
87 spherical particles. In these experiments the DEHS concentration in IPA was 0.03 % for generat-  
88 ing particles in the size range of 20 to 150 nm and 0.3% for 224 to 500 nm at laboratories  
89 A,B,C,D while 1% DEHS in IPA was used for the production of 10 to 150 nm and pure DEHS  
90 for particles above 150 nm was used at laboratory E. Indicative DEHS size distributions are pre-  
91 sented in the supplementary material. A diffusion dryer was used to ensure evaporation of the  
92 solvent. Alternatively a furnace was used to produce airborne silver particles in the size range of  
93 3-30 nm. A 99.99% silver slug was inserted in a boat inside the furnace which was then heated to  
94 800-1100 °C; the silver evaporated and the condensed into nanoparticles. Silver particles less than  
95 30 nm are compact and close to spheres. Almost no difference on filtration efficiencies was  
96 measured for 50 nm silver particles and sintered silver spheres (Kim et al., 2009). The particles  
97 were given the Boltzmann's equilibrium charging distribution by a krypton 85 or polonium 210  
98 neutralizer.

99 Laboratories A, B, C, D performed the experiments with monodisperse particle flow as  
100 the challenging aerosol, whereas laboratory E determined the filter media filtration efficiency by  
101 challenging it with polydisperse particles. Indicative set up schematics are presented in Figure 1.  
102 The monodisperse particles were obtained by classifying the aerosols from the generator using a

103 differential mobility analyzer (DMA). DMA accuracy was verified with polystyrene latex beads  
104 and the results are presented in the supplementary material (Table S1). The monodisperse parti-  
105 cles exiting the DMA mostly carried one electrical charge and were neutralized again by a neu-  
106 tralizer in order to minimize the filtration efficiency due to electrostatic forces. This approach re-  
107 duced the electrostatic effect in filtration and the associated uncertainties. Specimens of the sheet  
108 filter medium were fixed in the test filter holder and subjected to the test air flow corresponding  
109 to the prescribed filtration face velocity. Particles were counted upstream and downstream from  
110 the filter using either two condensation particle counters (CPCs) in parallel, or using only one  
111 such counter to measure the upstream and downstream concentrations alternately.

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

118 A pump positioned downstream drew the test aerosol through the test filter mounting as-  
119 sembly in both setups. Laboratory A, B, C and D used circular filter holders with diameter of 113  
120 mm, while laboratory E used a squared one with the length of the side 300mm. It should be noted  
121 that laboratory A used 38 mm diameter filter holder for the tests performed at 10 cm/s to reduce  
122 the required flow rate.

123 Six different filter media were tested. A wire mesh was tested because it is homogeneous  
124 and it could be used as a reference filter. Two bag filter media were also tested; F7 made of PET  
125 (polyethylene terephthalate) which is a charged filter and F7 made of glass which is an uncharged  
126 one. Finally, three highly efficient pleatable non charged filter media were tested; F9 and H13

127 glass fiber filters and E11 PTFE (polytetrafluoroethylene) synthetic filter. The summary of the  
128 different filter properties is presented in Table 2.

129           When the filter is charged, the filtration due to the electrostatic forces could be substantial.  
130 The surface potential of the filter could be reduced down to zero by exposing it to IPA vapor iso-  
131 propanol like Ohmi et al. (1994) and Xiao et al. (2014) mentioned. The purpose of the neutraliza-  
132 tion is two folds. Firstly, the minimum filtration efficiency could be tested when the filter medi-  
133 um is neutralized, so that the reported efficiency is a conservative value. Secondly, there is higher  
134 probability of variation if the filter samples are charged. Thus, the removal of the electrostatic  
135 charges from the medium improves the reliability of the test results.

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## 137 **3 Analyses**

### 138 **3.1 Filtration efficiency calculation**

139 The line losses for the upstream and downstream sampling might be different. The differ-  
140 ence can be significant when the particle size was very small and diffusion loss was important. In  
141 addition, some particles might be deposited at the inlet, outlet or walls of the filter holder. The  
142 upstream and downstream CPCs may have different counting efficiencies. Therefore it was im-  
143 portant to establish correlation ratios by performing the measurement without any filter medium  
144 in the filter holder. Then the filtration efficiency was measured with the filter placed inside the  
145 holder and calculated according to Wang and Tronville (2014).  
146

### 147 **3.2 Statistical analyses of the experimental results**

148 The reliability of the measurement method was verified by applying the statistical analysis  
149 according to ISO 5725-2. More details could be retrieved from the ISO 5725-2.  
150

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

$$156 \quad Y = m + b + e, \quad (1)$$

$$157 \quad s_L = \sqrt{\text{Var}(b)}, \quad (2)$$

159

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

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

where  $s_L$  is the between laboratory deviation.

Before determining the aforementioned deviations the outliers and stragglers were defined, using Mandel's h & k (Mandel J, 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.

### 3.3 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 calculat-



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

185

### 186 **3.4 Regression analysis**

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

192

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

193

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

194

195 Regression analysis provides estimates for the values of  $a$  and  $a_n$  coefficients. The inde-  
196 pendent 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.

### 197 **3.5 Correlation analysis**

198

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

### 204 **3.6 Determination of the figure of merit**

205

206 Hinds (1998), Dhaniyala and Liu (1999) and Wang et al. (2008) evaluated the filtration  
207 performance by obtaining by the figure of merit. The numerator is a measure of the filtration effi-

208 ciency, thus the higher the figure of merit the better the performance of the filter. The figure of  
209 merit could be calculated as:

210

$$211 \quad \gamma = -\ln(P / 100) / \Delta P \quad (6)$$

212

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

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## 216 **4 Results**

### 217 **4.1 Round robin test results analysis**

#### 218 **4.1.1 Filtration efficiency analysis**

219  
220 Five different laboratories tested the filtration efficiencies of different filter media at 2  
221 cm/s, 5 cm/s and 10 cm/s face velocity. Laboratories D and E did not participate in the tests using  
222 silver aerosols but E provided efficiency data down to 10 nm using DEHS diluted with IPA. La-  
223 boratory E used two different measurement principles to size the particles; thus, on the same effi-  
224 ciency curve for E both mobility diameter and optical diameter are presented, although no notice-  
225 able gap was observed between the sections corresponding to the two different instruments.  
226 Indicative results are presented in Figure 2. The rest of the results are presented in the supplemen-  
227 tary material. The theoretical filtration efficiency is presented with the thick solid line and the  
228 model equations are presented by Sachinidou et al. (2017). Diffusion was the main mechanism  
229 for the filtration efficiency below 100 nm. The smaller the particle was, the higher mobility it ac-  
230 quired, therefore, the filtration efficiency was high for small particles. In this size range, the effi-  
231 ciency dropped with the increasing face velocity due to the shorter time that the particle could  
232 diffuse. From 100 nm to 224 nm the main filtration mechanism was the interception and above  
233 this range impaction started to increase. Thus, the most penetrating particle size was around 224  
234 nm. The experimental results from laboratories A, B and C had low variances. The results from  
235 Laboratory D showed high within laboratory deviation, regardless the particle size or face veloci-  
236 ty, which could be attributed to a problem in their setup affecting the repeatability. Laboratory E  
237 often measured lower filtration efficiency compared to other laboratories.

238 In order to verify that the test procedure was reliable the experimental results were ana-  
239 lyzed according to section 3.2.

240 In order to acquire statistically reliable results, outliers and stragglers were defined for the  
241 tests using DEHS particles, for which all the five laboratories provided data therefore the sample  
242 number is five. The general mean filtration efficiency and the repeatability and reproducibility  
243 deviations, as fractions of the general mean filtration efficiency, were calculated and the results  
244 are presented in Table 3, Figure 3 and Figure 4 respectively. Increase of the face velocity slightly  
245 deteriorated the results leading to higher deviations. However, deviations strongly depended on  
246 the particle size. Filtration efficiency is a function of the particle size, thus close to the most pene-  
247 trating particle size (MPPS) the filtration efficiency is low and the experimental errors could sig-  
248 nificantly affect the measurement. Therefore, in this size range the deviations were higher.

249 The repeatability deviation is less than 0.1 and 0.03 of the absolute magnitude of the fil-  
250 tration efficiency for almost all the filters and particles sizes for DEHS and silver challenging  
251 particles respectively. When silver particles challenged the filter, the reproducibility deviation is  
252 below 0.05 for all the cases. For DEHS, the experimental results showed less than 0.2 deviation  
253 compared to the absolute magnitude of the filtration efficiency regardless the particle size or face  
254 velocity for all the filters apart from the wire mesh. Despite the fact that the absolute magnitude  
255 of reproducibility deviation for wire mesh was low (below 2.5% in most of the cases), the relative  
256 deviation was close to 0.2 at 5 cm/s and 0.3 at 10 cm/s because the filtration efficiency was low  
257 in the size range around the MPPS, thus, the experimental errors could impose a stronger effect in  
258 the measurement.

259 Overall, there were limited outliers (see supplementary material) for all the filters and in  
260 the whole particle size range regardless the challenging particle material. The repeatability devia-  
261 tion was mostly less than 0.1 of the absolute efficiency regardless the particle size, face velocity  
262 or challenging particle material. The only exception is for the wire mesh because filtration effi-  
263 ciency is low, thus, the error can highly contribute to the deviation. The reproducibility deviation

264 was more significant. Deviations for the silver particles were smaller compared to the DEHS be-  
265 cause only the three laboratories with the same setup participated. Also, the filtration efficiency is  
266 higher for smaller particles, thus, experimental errors are not so important compared to the abso-  
267 lute magnitude of the efficiency. Possible contributors of these deviations included the inhom-  
268 geneity of the filter media samples, different instruments and experimental setups in the partici-  
269 pating laboratories, inherent instrument uncertainties, etc.

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

278

#### 279 **4.1.2 Pressure drop analysis**

280 The pressure drop measured by the different laboratories is presented in Table 4. The wire  
281 mesh is a highly homogeneous filter, thus, the deviations among each laboratory measurements  
282 were low. Variation among the data for the other filters was observed. Laboratory A measured  
283 higher pressure drop compared to the other laboratories in many cases which could possibly be  
284 attributed to the measuring range of the instrument. The rest of the laboratories measured close  
285 pressure drop in almost all the cases. Variation among the results for the same filter could be par-  
286 tially explained by the filter inhomogeneity.

288

### 289 **4.1.3 Figure of merit of the tested filters**

290  
291 Inter-laboratory test results of the filtration efficiency and pressure drop were presented in  
292 Sections 4.1.1 and 4.1.2. The variations in the filtration efficiency and pressure drop may be at-  
293 tributed to the inhomogeneity of the filter samples. Inhomogeneity influences less the figure of  
294 merit than the efficiency or pressure drop, because if the inhomogeneity causes higher pressure  
295 drop for a specific piece of sample, then the corresponding efficiency should also be higher.

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

304

## 305 **4.2 Sensitivity Analysis**

### 306 **4.2.1 Particle Density effect on filtration efficiency**

307  
308 The particle density plays a role in determining the inertia of the particles and therefore  
309 the impaction and gravitational settling mechanisms. The effects are not substantial for nanopar-  
310 ticles.

311 Laboratories A, B and C carried out experiments for both DEHS ( $0.9 \text{ g/cm}^3$ ) and silver  
312 particles ( $10.49 \text{ g/cm}^3$ ) in the size range of 20 – 30 nm and the filtration efficiencies were com-  
313 pared to prove the aforementioned argument. The results showed that the DEHS and silver parti-

314 cles of the same electrical mobility size had almost the same filtration efficiencies, with the dif-  
315 ferences below 3%, thus the particle density did not notably affect the filtration efficiency in the  
316 size range well below 100 nm.

#### 317 **4.2.2 The effect of temperature and relative humidity on filtration efficiency**

318 The temperature affects the diffusion coefficient of the particles, therefore the filtration ef-  
319 ficiency. The variation was not expected to be wide since the round robin tests were at room con-  
320 ditions, therefore the effect was expected to be limited. In addition, the relative humidity affects  
321 the air density, thus, it might also affect the filtration efficiency. All the partner laboratories col-  
322 lected the relative humidity and temperature information during the round robin tests which al-  
323 lowed the comparison of the results measured at different relative humidity and temperature lev-  
324 els.  
325

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

336

337 **4.2.3 Effect of the concentration of the challenging aerosol on filtration effi-**  
338 **ciency**

339  
340 Concentration may affect the filtration efficiency depending on the range. If the challeng-  
341 ing aerosol concentration was too high, there could be particle agglomeration or loading effect on  
342 the filter which might affect the filtration efficiency like Kim et al. (2009) and Buha et al. (2013)  
343 stated. If in the proper range, the challenging aerosol concentration should not affect the filtration  
344 efficiency. It should be mentioned that high concentration could overload the particle sizer or in-  
345 crease the measurement errors from the particle counter due to the switch to photometric meas-  
346 urement mode.

347 Data from the partners during the inter-laboratory tests were analyzed to test if the chal-  
348 lenging concentration correlated with the filtration efficiency. The correlation coefficient was  
349 calculated using the correlation function in Excel. According to the results presented in the sup-  
350 plementary material, the absolute value of the correlation coefficient was in almost all the cases  
351 smaller than 0.5, thus, there was no clear indication that the challenging particle concentration  
352 affected the filtration efficiency in the range of tens to tens of millions particles per cc.

353  
354 **4.2.4 Challenging particle size distribution, SAFR and neutralization efficien-**  
355 **cy**

356  
357 When the DMA is used as a classifier, its set parameters should be studied to ensure the  
358 minimization of the measurement artifacts. Sheath to aerosol flow ratio (SAFR) could affect the  
359 monodispersity of the challenging particle flow based on the generated particle size distribution.  
360 According to Sachinidou et al. (2017) a SAFR above or equal to five could minimize the artifacts  
361 due to this factor for the investigated filtration tests and the particle size distribution at the DMA  
362 inlet would have marginal effect on the measurement. In addition, neutralization efficiency for



363 the particles entering the DMA was crucial so as to avoid bigger multiply charged particles to en-  
364 ter the challenging particle flow.

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

369

#### 370 **4.2.5 Flow distribution**

371 Flow distribution depends on the filter holder geometry, thus the filter holder size and  
372 shape could possibly affect the flow velocity distribution.

373 The test filter media with different surface areas may possess different uniformity levels  
374 thus leading to variation in the test results. Laboratories A and B tested the wire mesh filter me-  
375 dia, which is a highly homogeneous filter, at 5 cm/s using two different filter holders ( $D_{\min}=38$   
376 mm and  $D_{\max}=113$  mm). The results presented in Figure 6 showed a good agreement among the  
377 filtration efficiencies measured with different filter holders in the whole particle size range for the  
378 wired mesh, thus, indicating no obvious link between the filter surface area and filtration effi-  
379 ciency if the filter medium was highly uniform. Similar results were presented by Sachinidou et  
380 al. (2017) supporting that the face velocity distribution was homogeneous upstream the filter me-  
381 dia.

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

#### 387 **4.2.6 Charge on the filter**

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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 figure 7 and it is crucial for particles above 30 nm.

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## 395 **5 Conclusions**

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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 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.

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

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

430

### 431   **Acknowledgement**

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433   zation activities regarding nanotechnologies and nanomaterials.”

434

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520

521 **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<br>Constant<br>output<br>atomizer | TSI Nanoscan SMPS<br>3910 & PMS LAS-X<br>II (TSI 3340) |
|---|------|--|--|

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**Table 2 Filters used in the interlaboratory tests**

| filter class: | filter type: |           | media type: |         |                            |             |               |
|---------------|--------------|-----------|-------------|---------|----------------------------|-------------|---------------|
|               | bag filter   | pleatable | synthetic   |         |                            | glass fiber | PEI synthetic |
|               |              |           | non-charged | charged | discharged/<br>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           |               |

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**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)    | 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           | Filtration efficiency (%) - Silver |      |        |      |          |      |      |      |      |      |       |       |

| size (nm) |      |     |       |     |       |     |       |     |       |       |       |       |
|-----------|------|-----|-------|-----|-------|-----|-------|-----|-------|-------|-------|-------|
| 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 |

527

528 **Table 4 Average filter media pressure drop measured by the different labor. toris.**

| Pressure drop (Pa) |                 |     |     |     |     |     |
|--------------------|-----------------|-----|-----|-----|-----|-----|
|                    | Velocity (cm/s) | A   | B   | E   | D   | C   |
| wire mesh          | 5               | 57  | 54  | 55  | 51  | 58  |
|                    | 10              | 112 | 108 | 112 | 102 | 112 |
| F7 PET Charged     | 5               | 36  | 22  | 21  | 25  | 23  |
|                    | 10              | 53  | 46  | 37  | 46  | N/A |
| F7 glass           | 5               | 32  | 25  | 21  | 21  | 26  |
|                    | 10              | 51  | 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 |

529

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

| 5 cm/s |           |     |        |          |     |     | 10 cm/s   |     |          |      |      | 2 cm/s |  |
|--------|-----------|-----|--------|----------|-----|-----|-----------|-----|----------|------|------|--------|--|
| 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 |
| 150nm |           |     |        |          |     |     |           |     |          |      |     |
|       | 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 |

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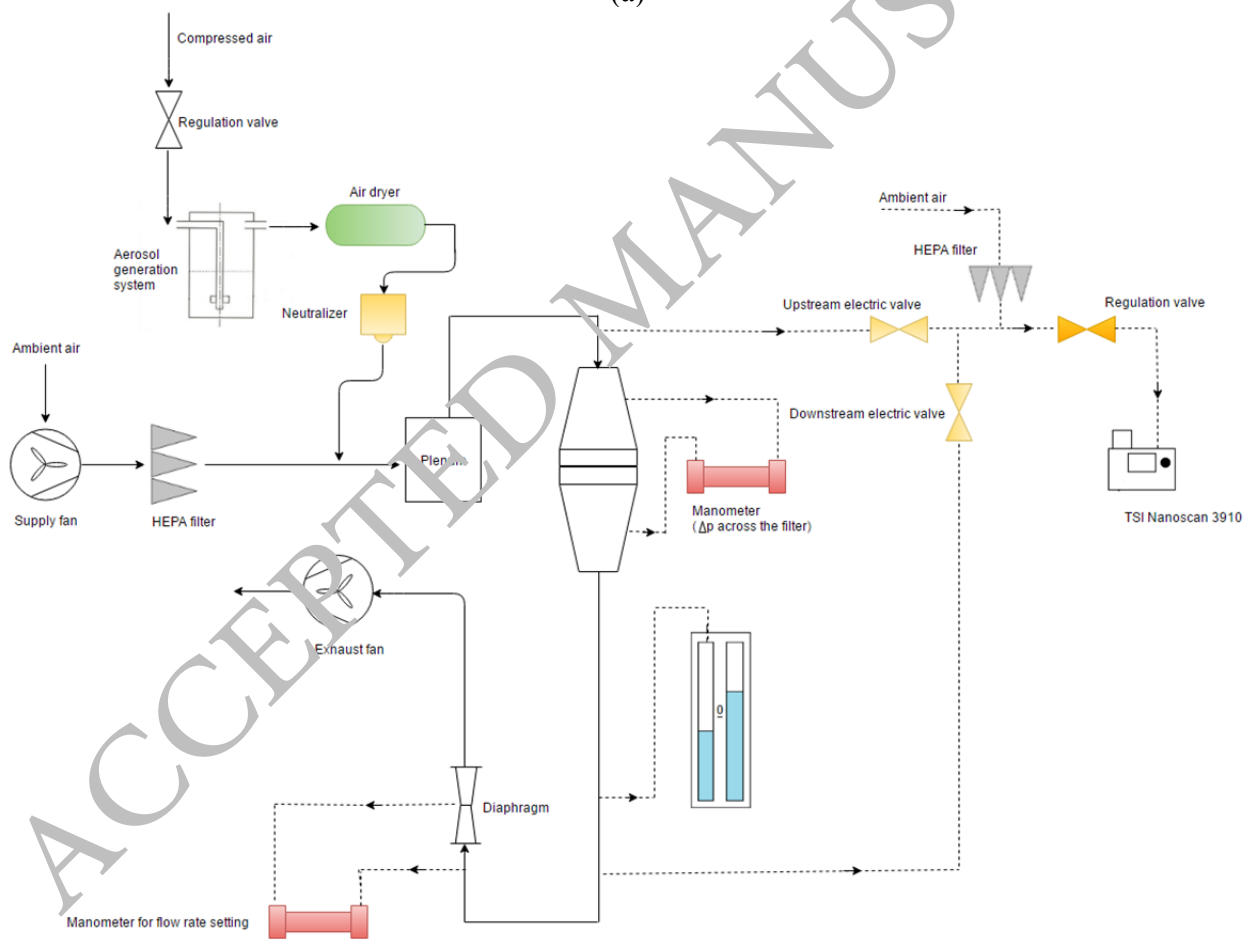
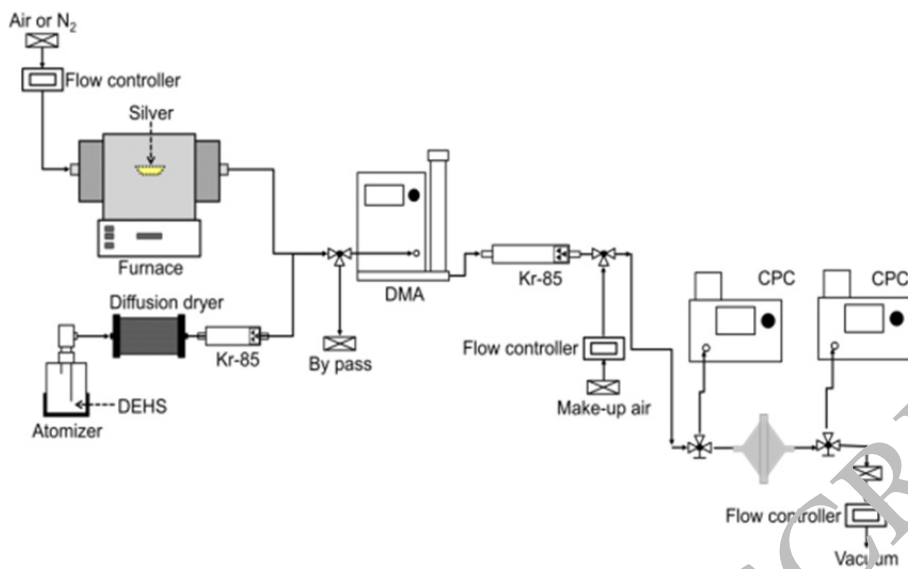
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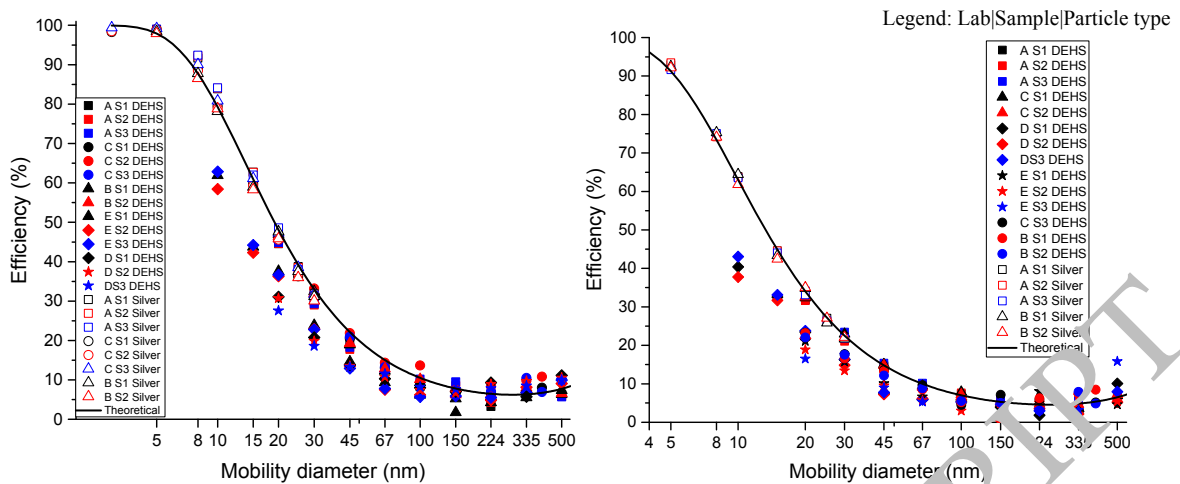
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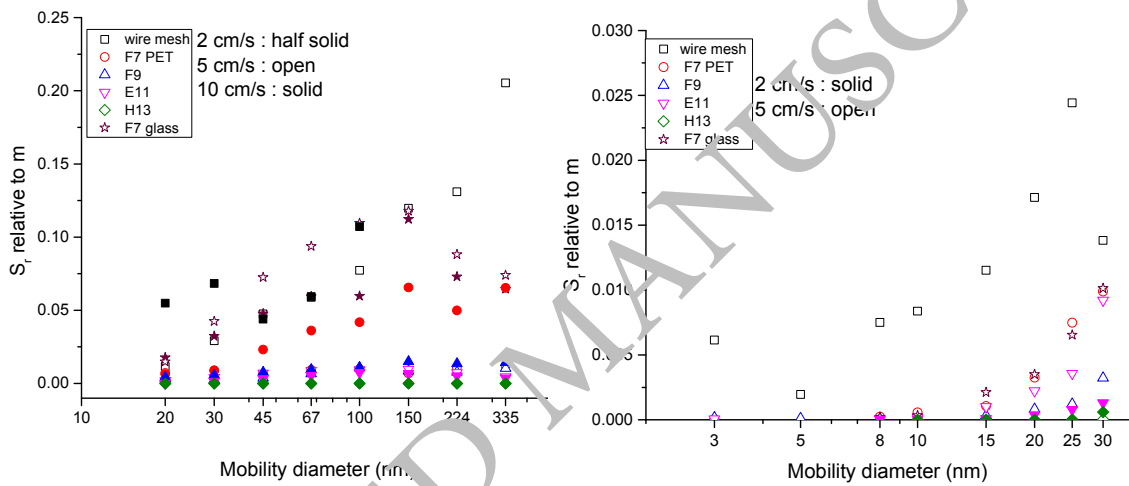


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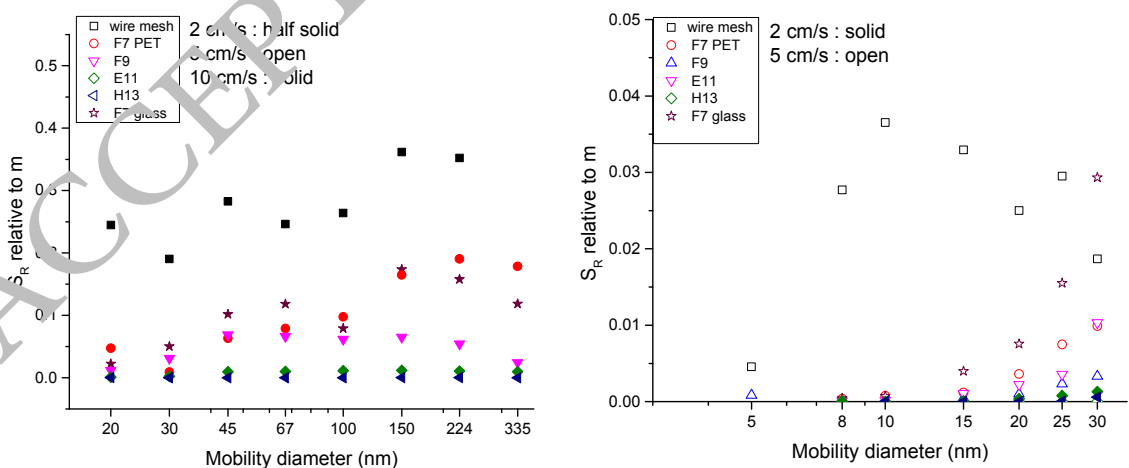
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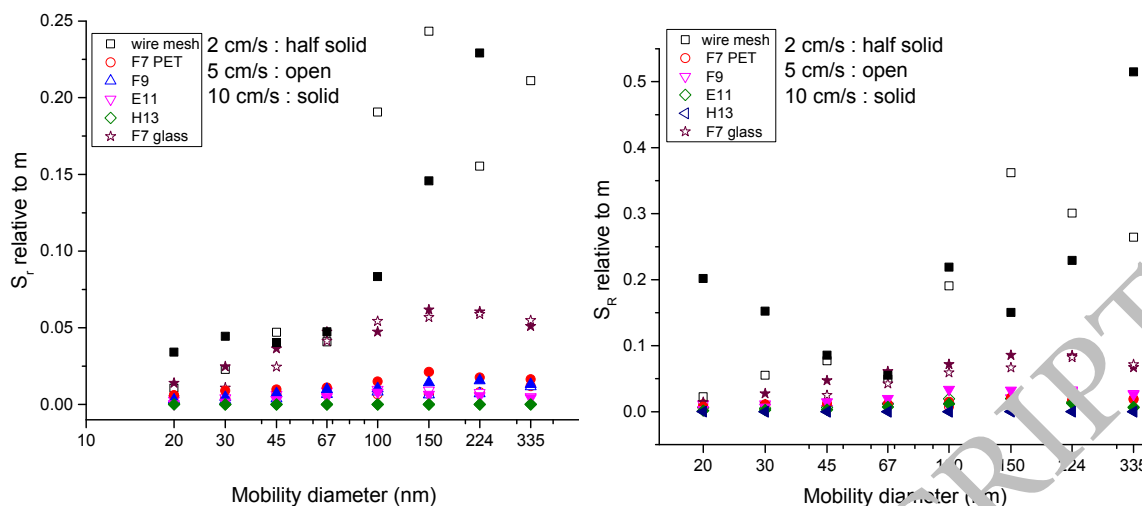
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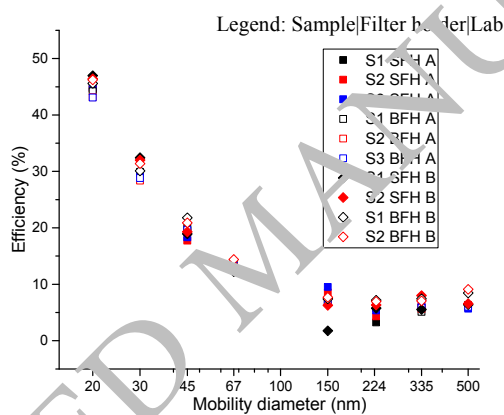
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554 silver in the right panel).



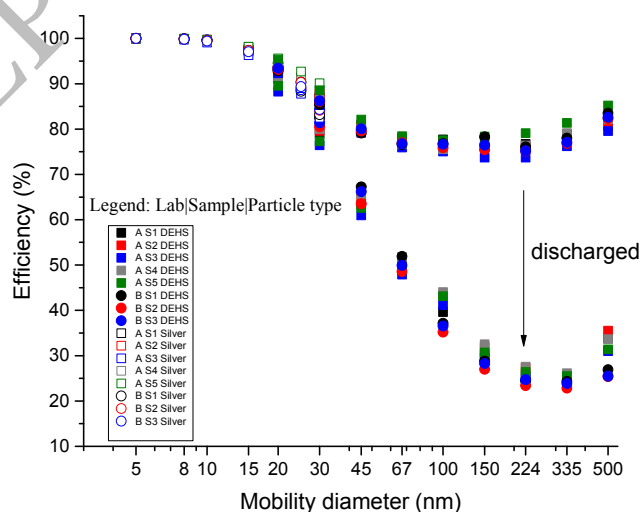
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558  
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