Inter-laboratory tests of the methodology for filtration efficiency tests in different filter media against nanoparticles

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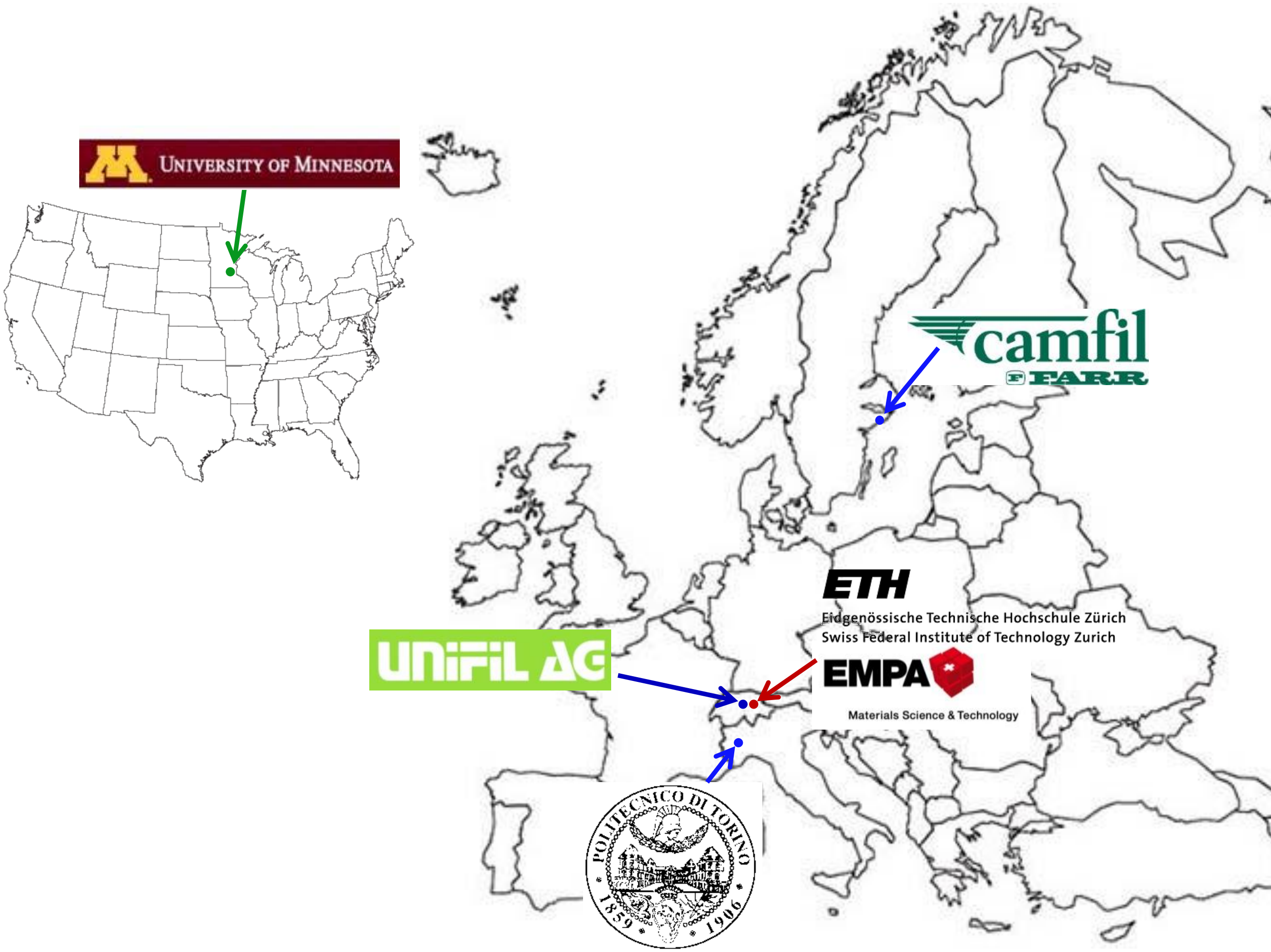
Outline

- Project background
- Pre-normative research
- Qualification of the setup
- Inter-laboratory tests
- Summary

Project consortium

Methodology to Determine Effectiveness of Filtration Media against Nanoparticles in the Size Range of 3 to 500 Nanometer



Reference lab
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Prof. Jing Wang

Supporting lab
Particle Technology Lab
University of Minnesota, USA
Prof. David Y.H. Pui

Round-robin test labs
Camfil, Sweden
Mr. Mikael Eriksson
Politecnico di Torino, Italy
Prof. Paolo Tronville
Unifil, Switzerland
Mr. Nägeli Andreas

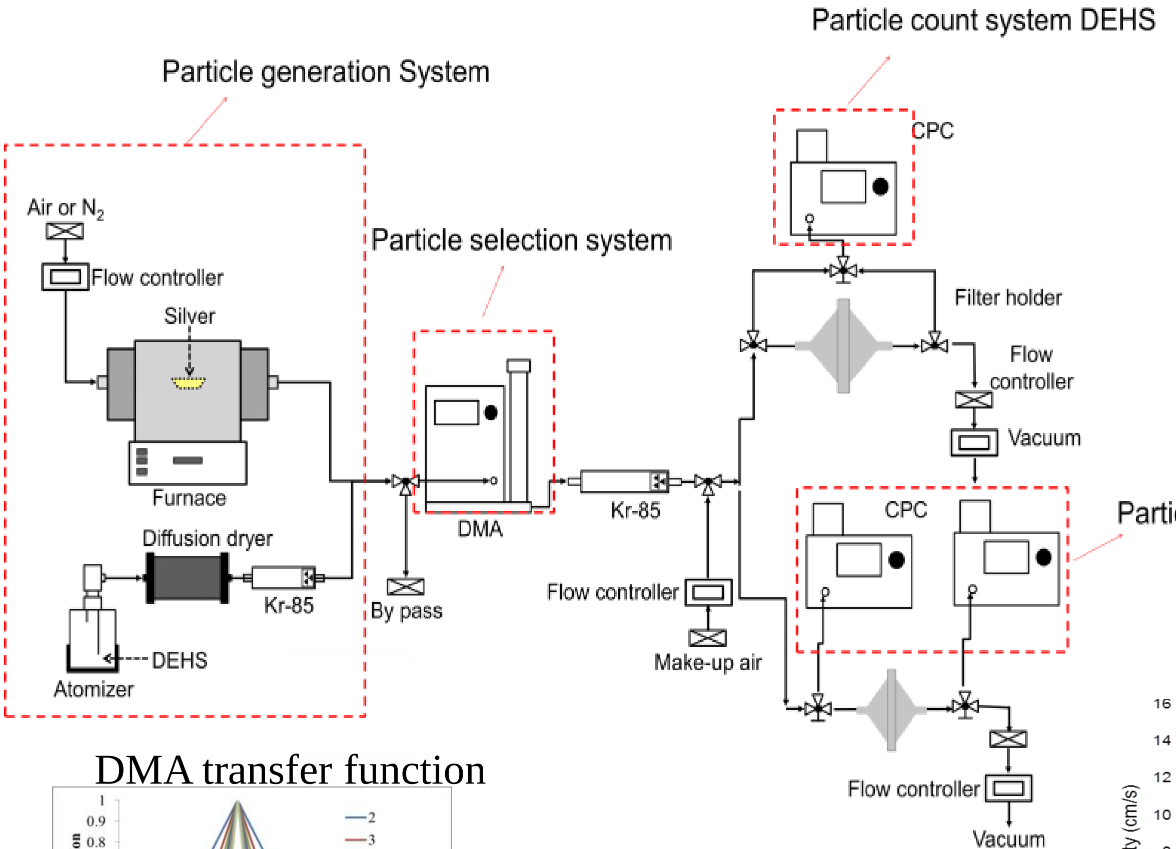
Summary of relevant air filtration standards

Background
Pre-norm Research
Qualification
Interlaboratory Tests

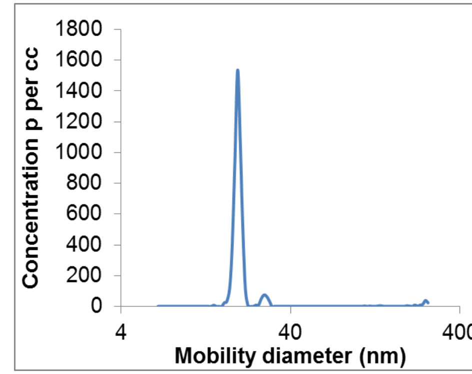
Designation	Title	Test particle	Remark
ANSI/ASHRAE Standard 52.2 (2012)	Method of testing general ventilation air-cleaning devices for removal efficiency by particle size	KCl particles in the range of 0.3–10 µm	Wind tunnel test using optical or aerodynamic particle sizers
EN 779 (2012)	Particulate air filters for general ventilation—determination of the filtration performance	DEHS particles in the range of 0.2–3.0 µm	Wind tunnel test using optical particle sizers
ISO 29463 series (2011a, b, c, d, e)	High-efficiency filter and filter media for removing particles in air	DEHS, PAO, and Paraffin Oil in the range 0.04 µm to 1.0 µm (0.1–2.0 µm with OPS)	Focus on the minimum efficiency at the MPPS and local efficiencies
NIOSH 42 CFR 84.181 (1995)	Non-powered air-purifying particulate filter efficiency level determination	A mass median aerodynamic diameter of ~0.3 µm, NaCl or DOP polydisperse particles	For respirator certification
EN 1822 series (2009a, b, c, d, e)	High-efficiency air filters (EPA, HEPA and ULPA)	DEHS, PAO, and Paraffin Oil in the range 0.05 µm to 0.8 µm (0.1–2.0 with OPS)	Focus on the minimum efficiency at the MPPS and local efficiencies
EN 143:2000	Respiratory protective devices—Particle filters—requirements, testing, marking	Various aerosol allowed including sodium chloride and paraffin oil	For respirator air filter certification
ISO 29461-1:2013	Air intake filter systems for rotary machinery—test methods—part 1: static filter elements	DEHS particles in the range of 0.3–3.0 µm	Wind tunnel test using optical particle sizers

Jing Wang & Paolo Tronville (2014), Toward standardized test methods to determine the effectiveness of filtration media against airborne nanoparticles, J Nanopart Res 16:2417

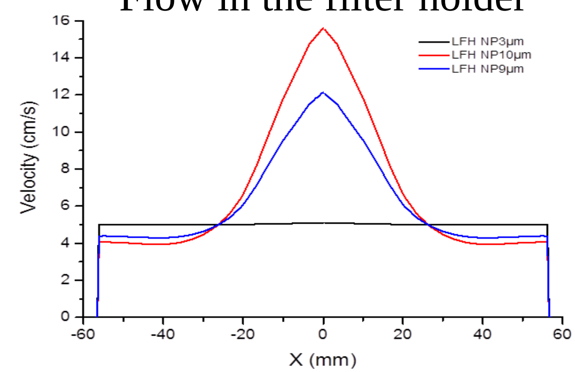
Filtration tests



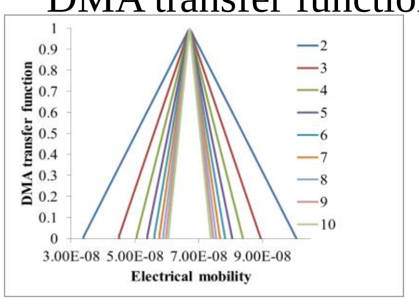
Particle size



Flow in the filter holder

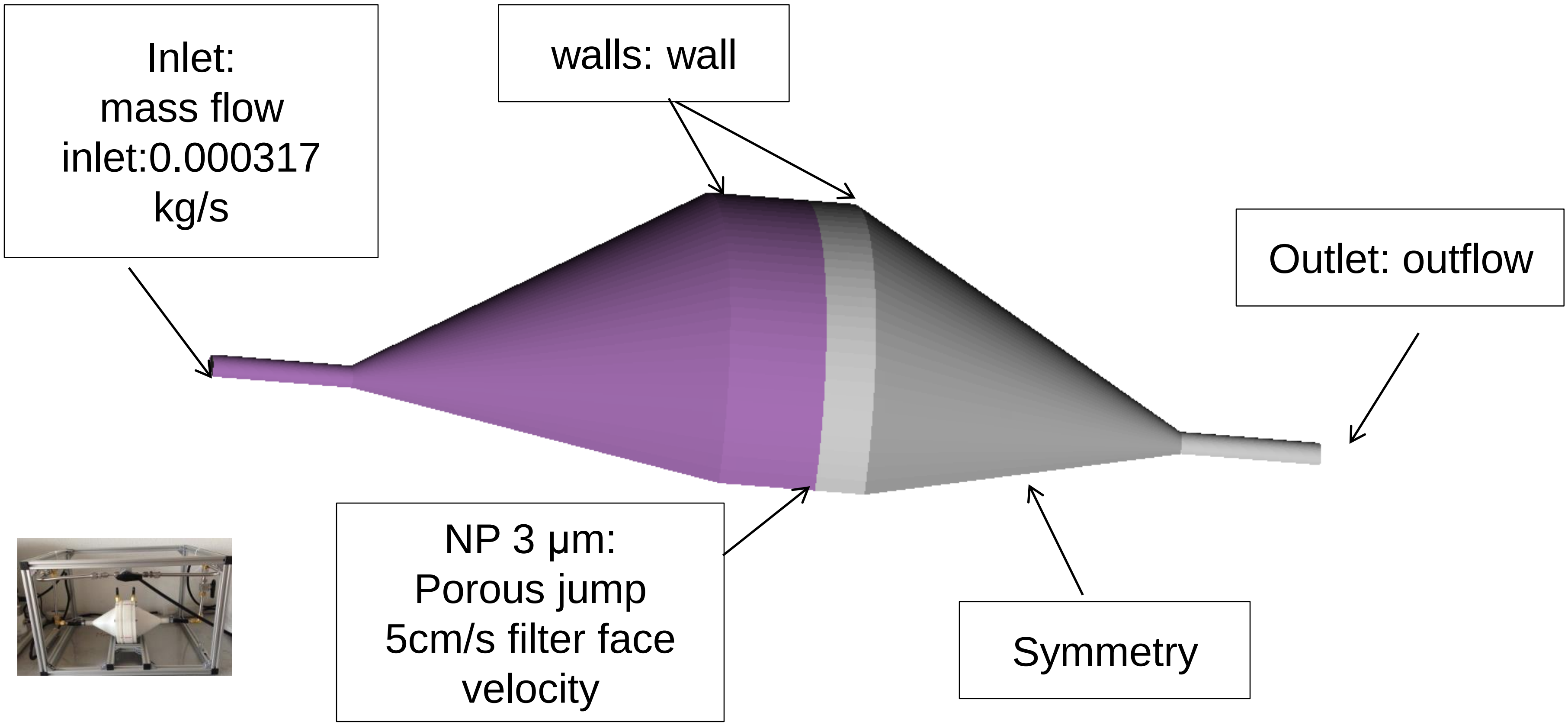


DMA transfer function



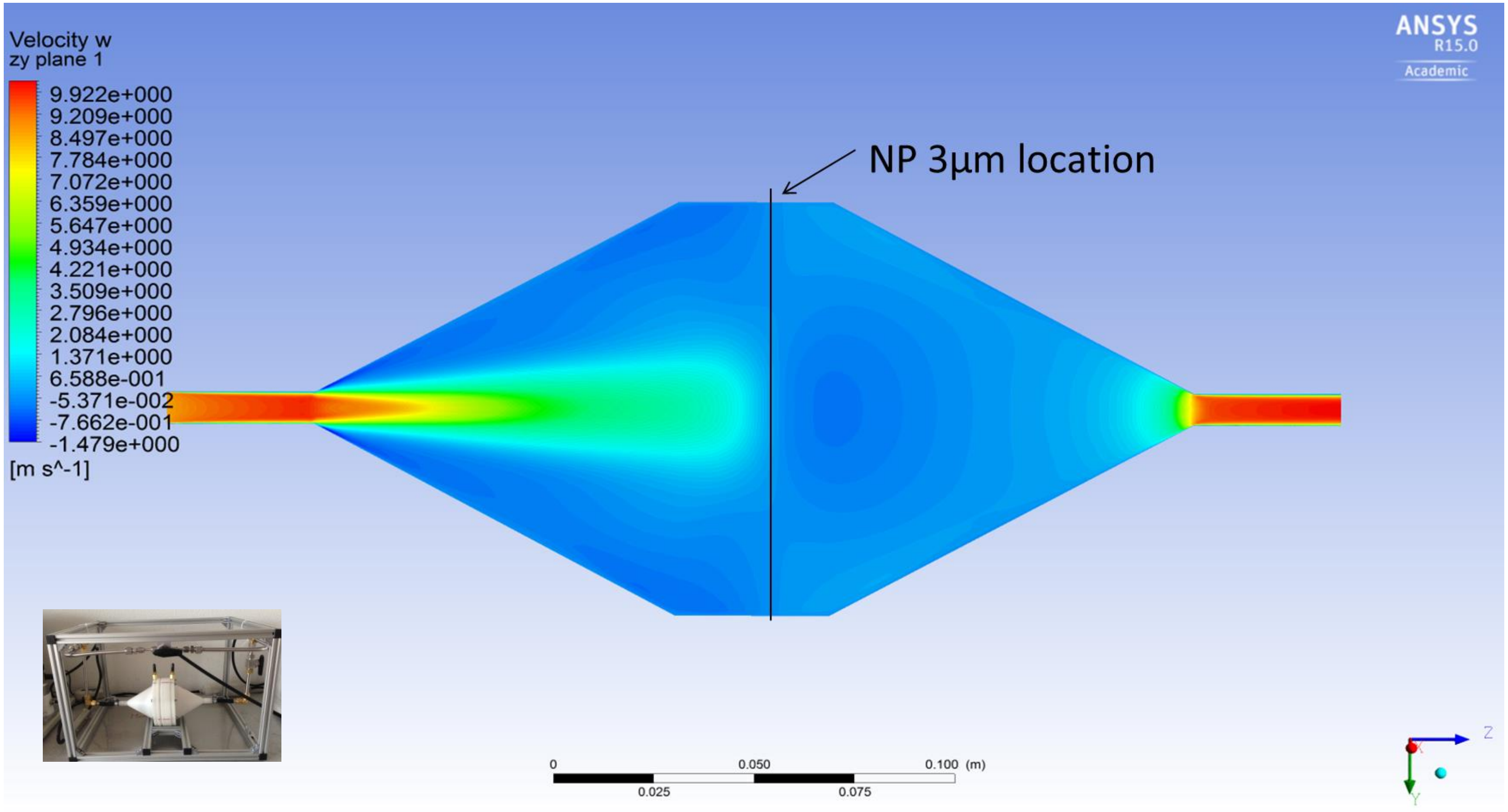
Sachinidou, P., Bank, Y.K., & Wang, J, Aerosol Sci & Tech, 2016

Flow distribution-CFD analysis



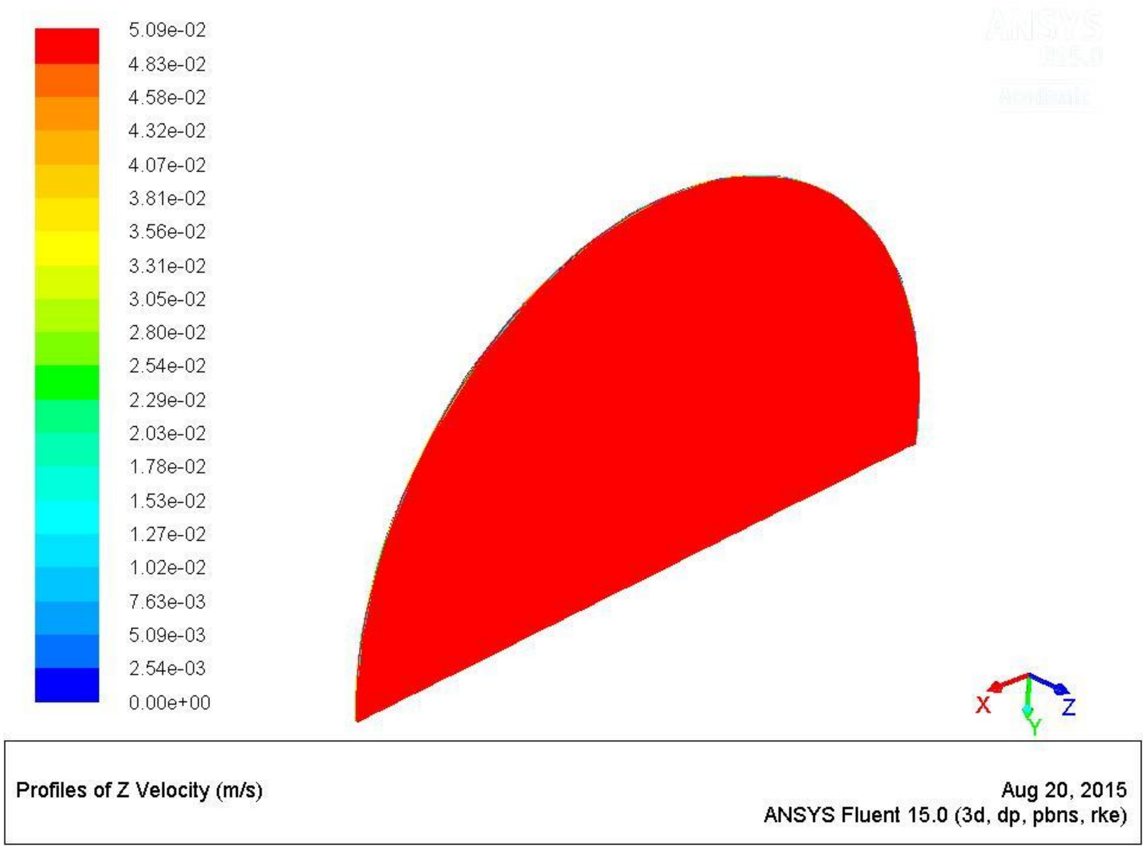
- Flow distribution in the large filter holder was simulated using ANSYS FLUENT.
- K-ε realizable model was applied and mesh independency study was performed.
- NP 3μm filter which is homogeneous was chosen for the investigation and simulated with porous jump boundary conditions.

Flow distribution- velocity distribution

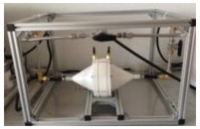


Face velocity has a jet profile which is distributed homogeneously before the filter.

Flow distribution-face velocity distribution immediately upstream the filter

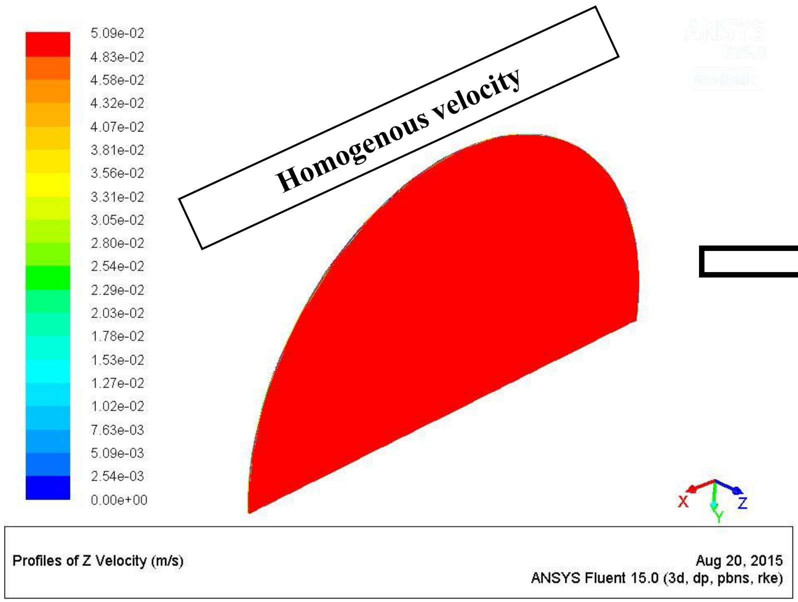


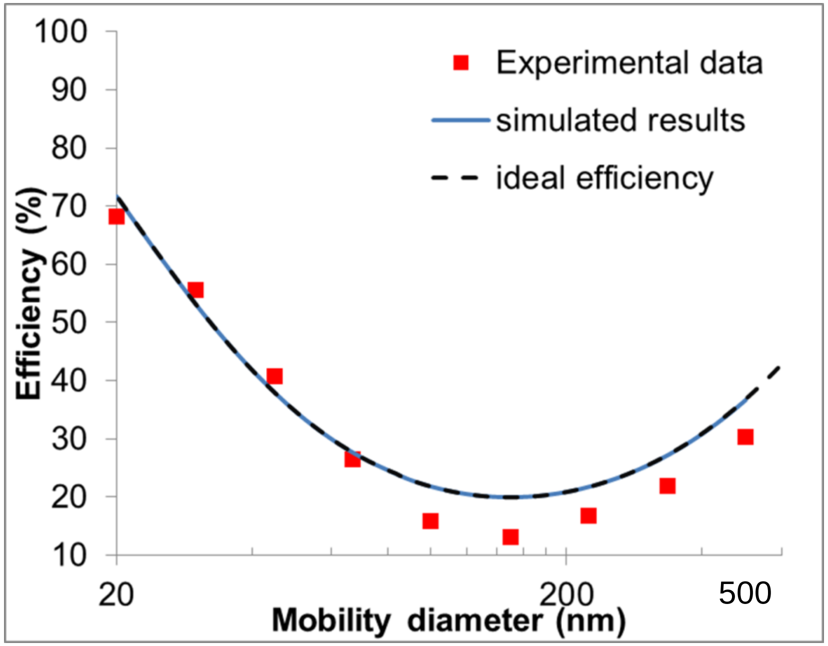
Face velocity is homogeneously distributed upstream the filter



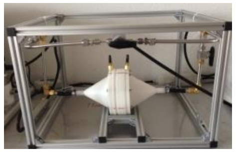
Monodispersity investigation

- Flow distribution incorporated in the filtration model





- Flow distribution does not affect the calculated filtration efficiency.



Qualification procedure

- **Zero count test**
- **Counting accuracy calibration**
- **DMA test**
- **Neutralization efficiency test**
- **Zero efficiency test**
- **Preparatory checks**

Qualification of the test rig -Neutralization efficiency

- **Neutralization Test**

The neutralization effectiveness of the neutralizer was checked using two DMA connected in series. The first one was used to pre-select the desired particle diameter and the second one was used to select the particle diameter corresponding to singly, doubly and triply charged particles. This set up allows checking the efficiency of the neutralizer that is located inside the second DMA. The experimental particle charge ratio was compared with the theoretical one (Wiedensohler (1988) and Kim et al. (2005)) The same experiments were carried, using an additional neutralizer in between the two DMA in order to study if the residence time does not affect the neutralization efficiency.

Qualification of the test rig -Neutralization efficiency

Background

Pre-norm Research

Qualification

Interlaboratory Tests

			1charges/icharge	
	Mobility diameter (nm)	Raw counts	Experimental Ratio	Theoretical Ratio
1 charge	**51.4**	706.53		
2 charges	35.66	26.97	26.19	24.2

			1charges/icharge	
	Mobility diameter (nm)	Raw counts	Experimental Ratio	Theoretical Ratio
1 charge	**95.6**	190.07		
2 charges	64.99	22.87	8.31	7.23

			1charge/icharge	
	Mobility diameter (nm)	Raw counts	Experimental Ratio	Theoretical Ratio
1 charge	**193.3**	84.31	1	
2 charges	125.7	26.42	3.19	2.94
3 charges	99.22	4.855	17.36	14.49

ETH (Kr-85)

			1charges/icharge	
	Mobility diameter (nm)	Raw counts	Experimental Ratio	Theoretical Ratio
1 charge	**33.98**	22389.4		
2 charges	23.73	277.8	80.68	78.06

			1charges/icharge	
	Mobility diameter (nm)	Raw counts	Experimental Ratio	Theoretical Ratio
1 charge	**80.58**	7246		
2 charges	55.28	784	9.24	9.62

			1charge/icharge	
	Mobility diameter (nm)	Raw counts	Experimental Ratio	Theoretical Ratio
1 charge	**191.1**	4690.8		
2 charges	124.9	1455	3.22	2.98
3 charges	98.6	342.8	13.66	14.86

UMN (Po-210)

- Results show the experimental ratio is in good agreement with the theoretical one.

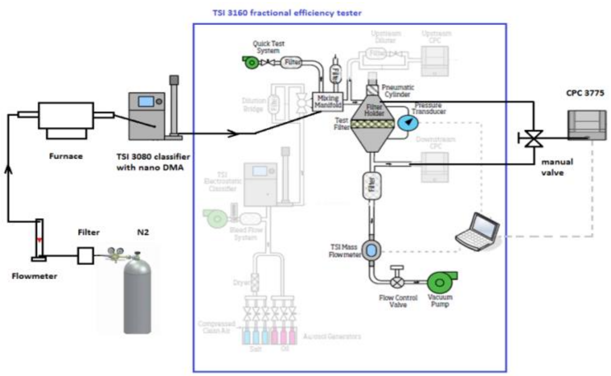
Test setup

Background

Pre-norm Research

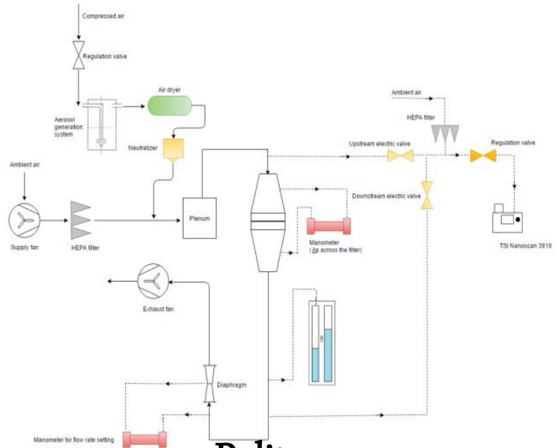
Qualification

Interlaboratory Tests

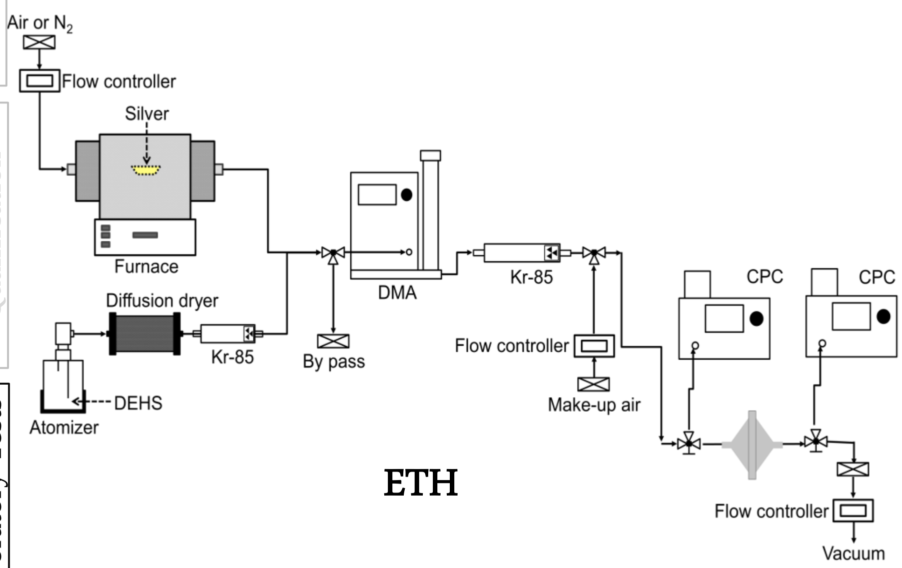


Unifil

Camfil



Polito

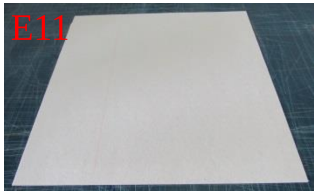


ETH

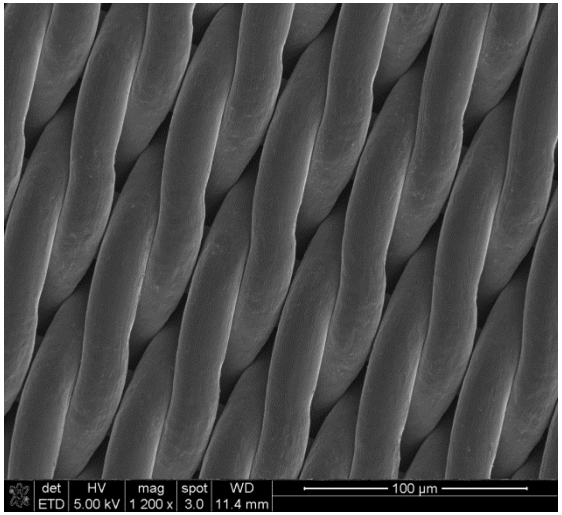
UMN

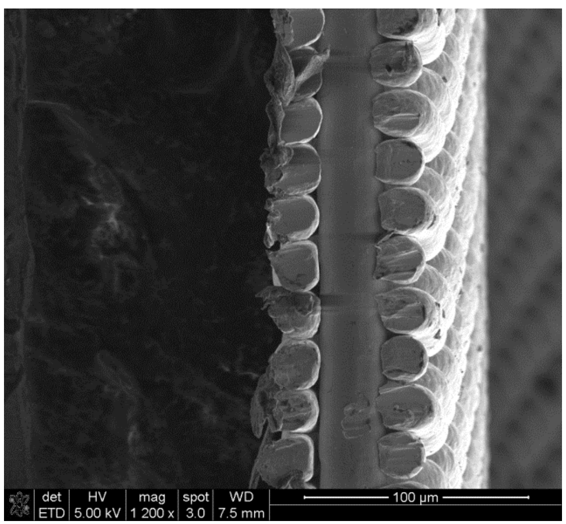
Filters tested

filter class:	**filter type:**		media type:				
	bag filter	pleatable	synthetic			glass fiber	PTFE Synthetic
			non-charged	charged	discharged/ non charged		
Mesh			X				
M5	X			X			
NP 3µm			X				
F7	X			X	X		X
F7	X		X			X	
F9		X	X			X	
E11		X	X				X
H13		X	X			X	

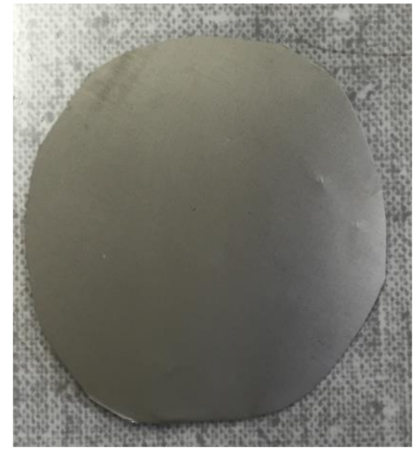


Interlaboratory Tests: Twilled Dutch weave mesh 350x2600



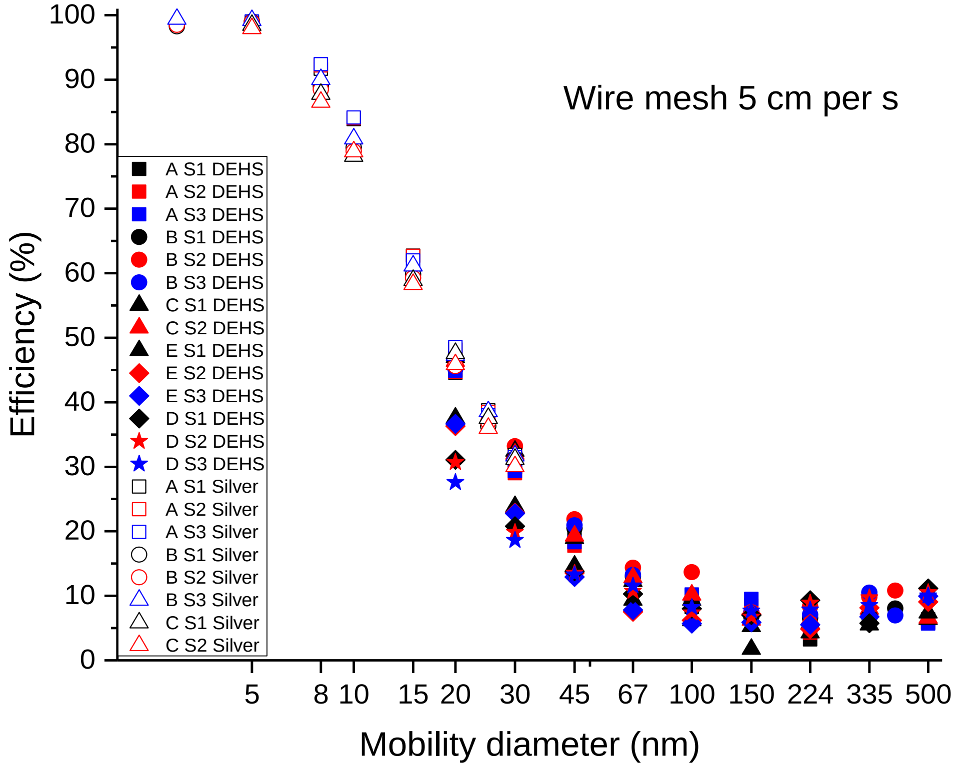


SEM image



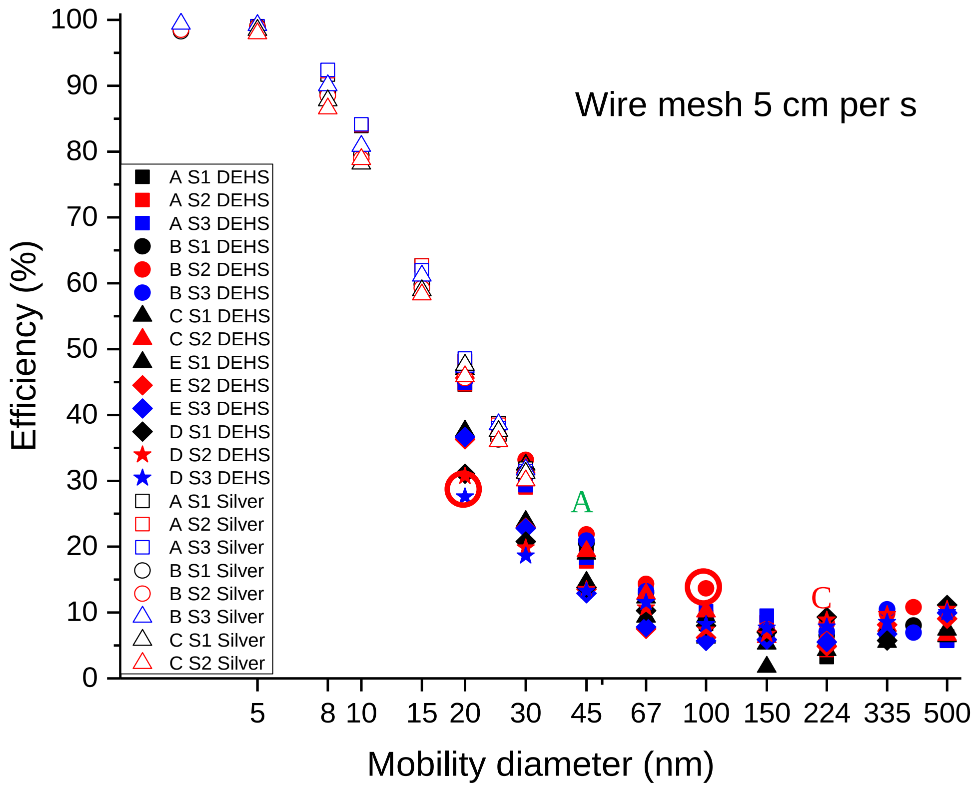
Twilled Dutch weave 350x2600		
Solidity	0.62	**-**
Fiber Size (wrap)	32	**µm**
Fiber Size (weft)	22	**µm**
Filter thickness	0.08	**mm**
Material	Stainless Steel	

Wire mesh



- Filtration efficiency results are in accordance with each other among the difference laboratories

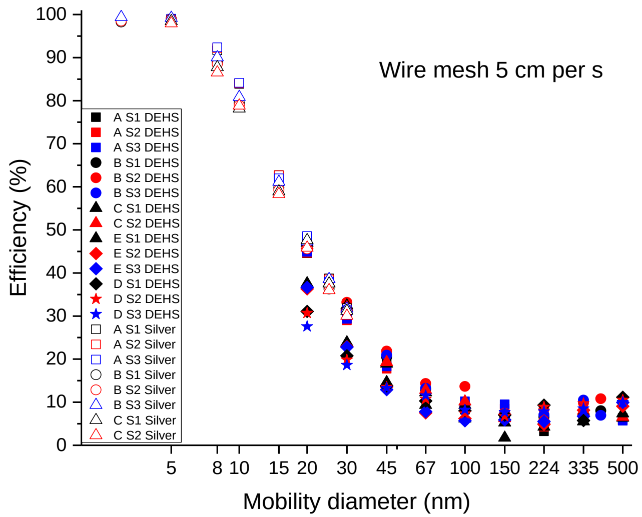
Wire mesh



Green Color: stragglers
Red Color: outliers

- There are not many straggles or outliers in the whole particle size range.

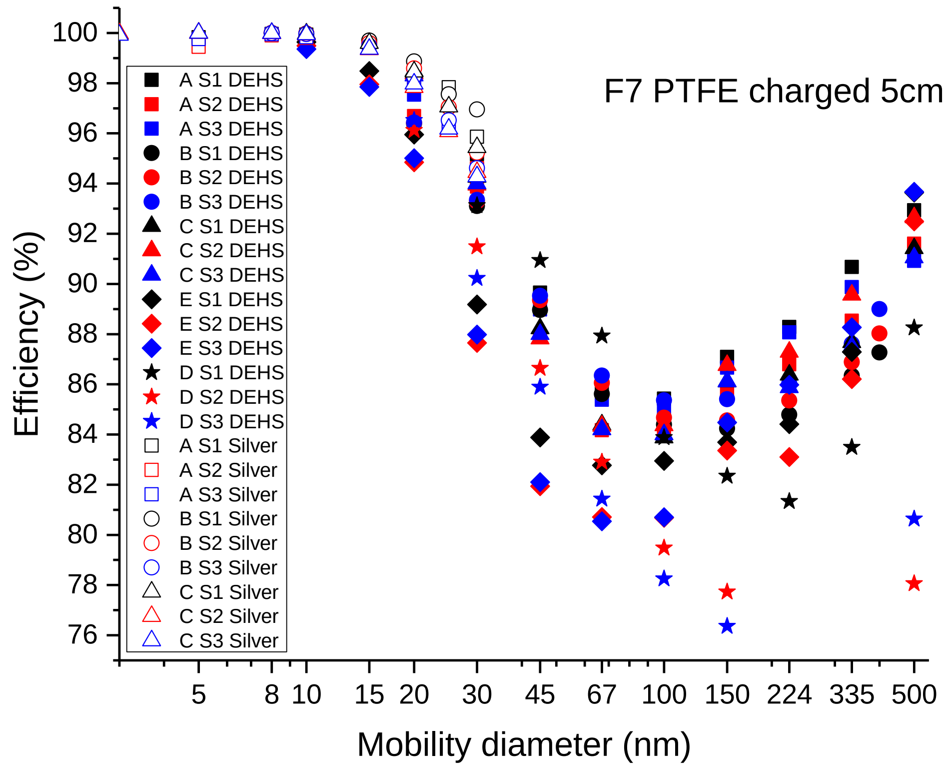
Wire mesh



	m	S_r	S_l	S_R
20	43.2	0.5	4.6	4.6
30	27	0.79	5.59	5.64
45	17	0.81	3.48	3.57
67	11.653	0.685	2.252	2.354
100	8.087	0.625	1.618	1.735
150	7.18	0.86	0.77	1.15
224	6.26	0.82	1.78	1.96
335	7.55	1.55	0.55	1.64

- The results shows small variance; Thus, statistical analysis reveals a few stragglers or outliers.
- The variances calculated according to the statistical analysis are low for almost all the particle size range.

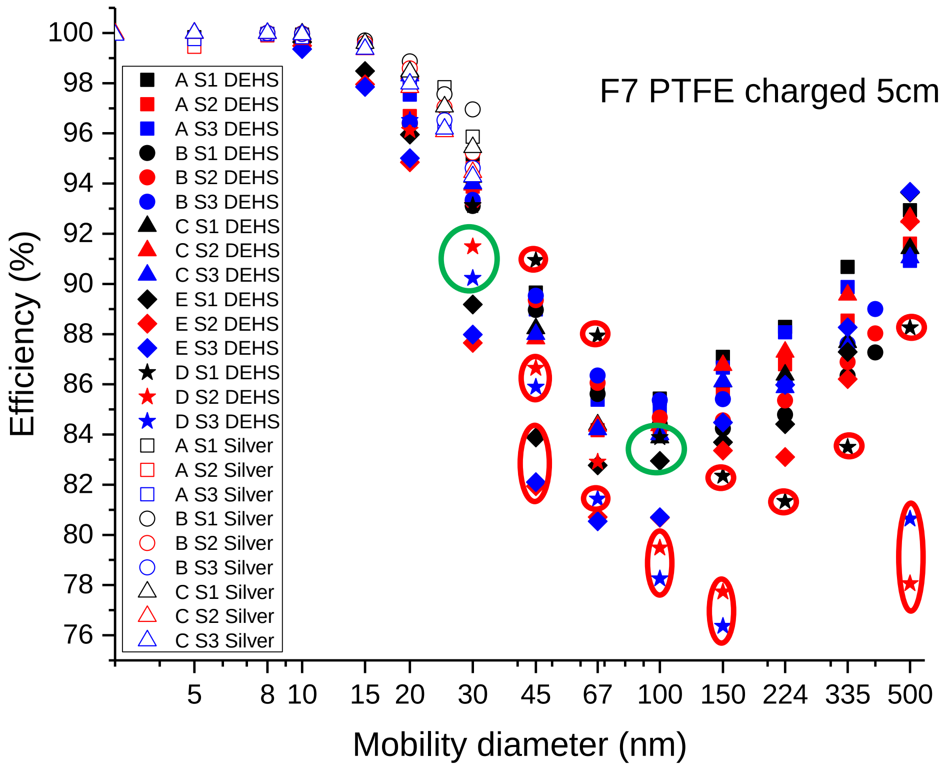
F7 charged



	Pressure drop (Pa)				
	A	E	B	D	C
S1	34	20	22	27	25
S2	37	21	23	23	21
S3	36	22	23	25	21

- D and E measures smaller efficiency compared to the ones measured by the other labs.
- Pressure drop is close among the different laboratories except from A. Possibly this could attributed to the measurement range of the instrument at laboratory A (minimum limit equals to 13Pa).

F7 charged

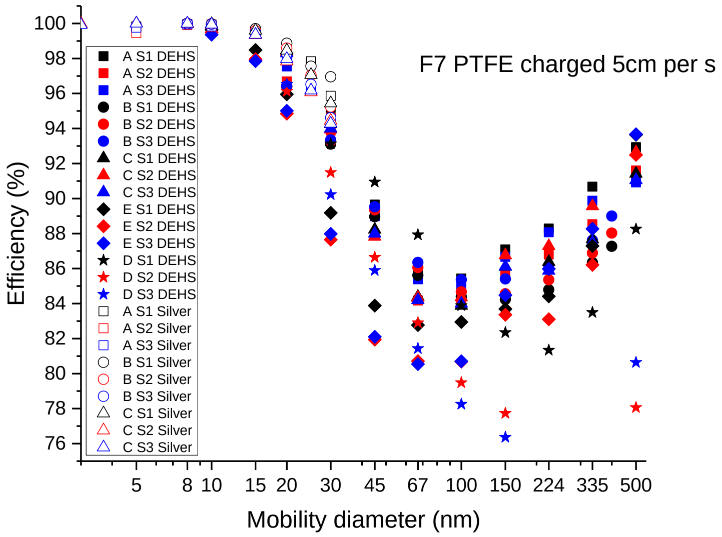


	Pressure drop (Pa)				
	A	E	B	D	C
S1	34	20	22	27	25
S2	37	21	23	23	21
S3	36	22	23	25	21

Green Color: stragglers
Red Color: outliers

- High variance for laboratory D; Statistical analysis reveals many stragglers and outliers for laboratory D.

F7 charged

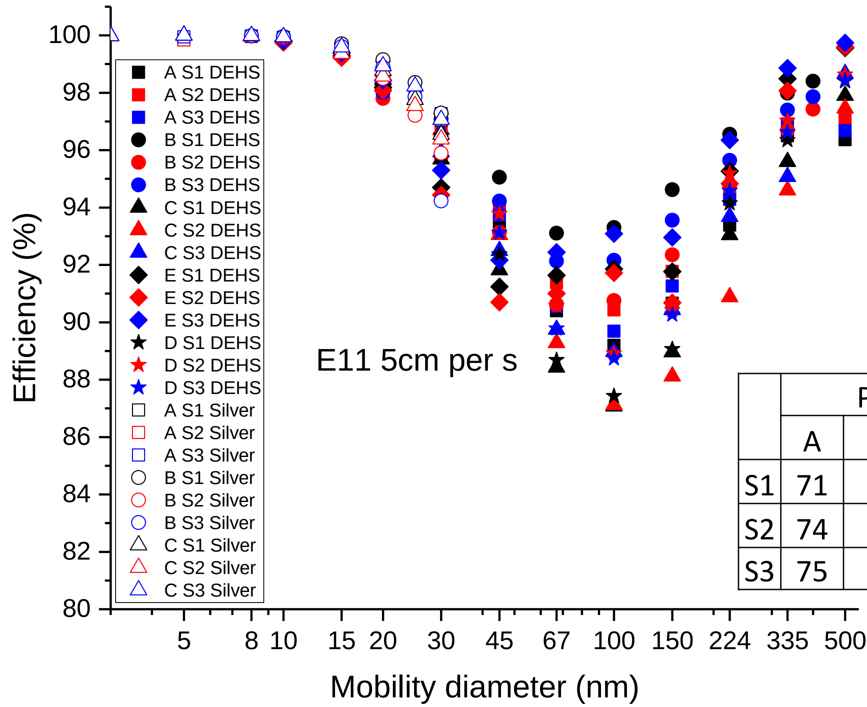


	Pressure drop (Pa)				
	A	E	B	D	C
S1	34	20	22	27	25
S2	37	21	23	23	21
S3	36	22	23	25	21

	m	S_r	S_l	S_R
20	96.8	0.7	1.1	1.3
30	92.22	0.85	2.53	2.67
45	88.80	0.61	0.48	0.77
67	84.189	0.836	2.066	2.228
100	83.760	0.859	1.592	1.809
150	85.26	0.60	1.24	1.38
224	85.99	0.97	1.33	1.65
335	88.02	1.01	1.14	1.52

- Variances are about 1 – 2 %.

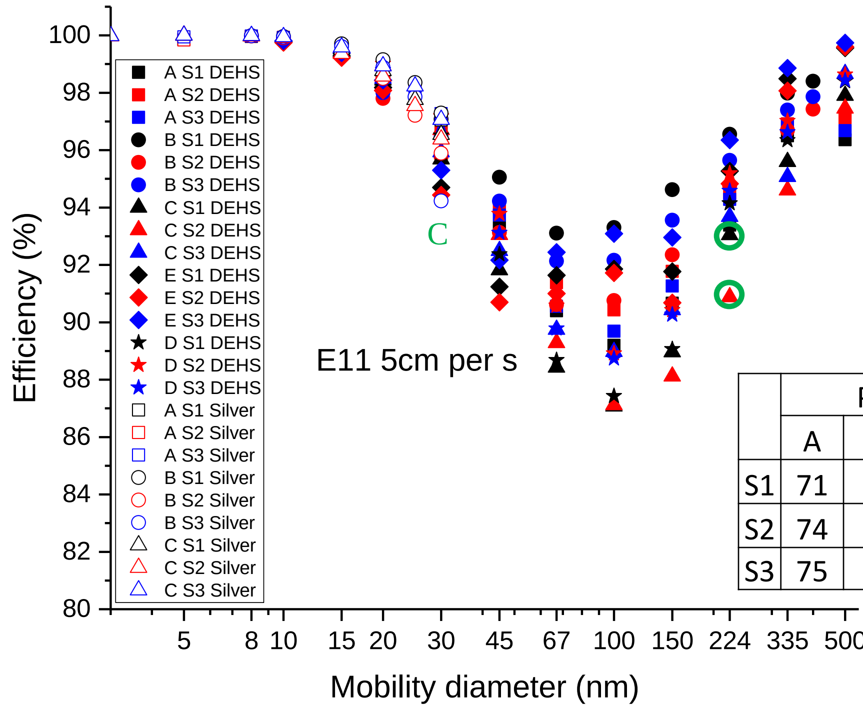
E11 (5cm/s)



	Pressure drop (Pa)				
	A	E	B	D	C
S1	71	76	62	57	54
S2	74	78	57	53	50
S3	75	80	59	60	66

- The deviation in filtration efficiency is low among the different laboratories.
- There is a deviation in pressure drop measured among the different laboratories.

E11 (5cm/s)

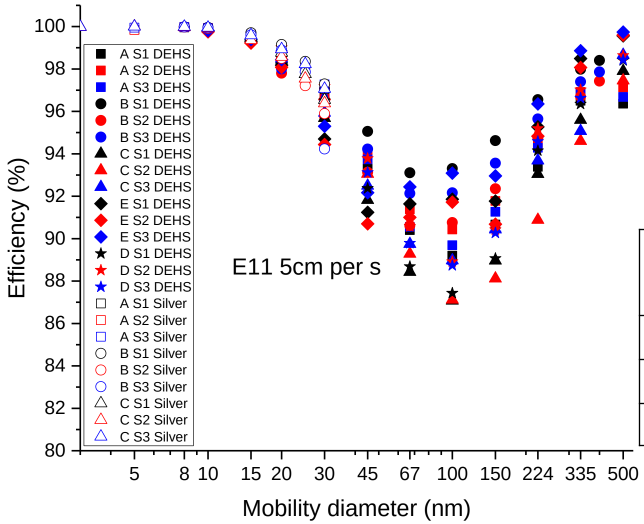


	Pressure drop (Pa)				
	A	E	B	D	C
S1	71	76	62	57	54
S2	74	78	57	53	50
S3	75	80	59	60	66

Green Color: stragglers
Red Color: outliers

- The deviation in filtration efficiency is low among the different laboratories. Thus, statistical analysis does not reveal outliers.

E11(5cm/s)



	m	S_r	S_l	S_R
20	98.4	0.3	0.2	0.4
30	96.22	0.48	0.83	0.96
45	92.98	0.74	1.02	1.26
67	90.759	0.877	1.056	1.373
100	90.196	0.849	1.962	2.138
150	91.27	0.90	1.56	1.80
224	94.61	0.85	1.05	1.35
335	97.00	0.47	1.11	1.21

	Pressure drop (Pa)				
	A	E	B	D	C
S1	71	76	62	57	54
S2	74	78	57	53	50
S3	75	80	59	60	66

- The deviation in filtration efficiency is low among the different laboratories. Thus, statistical analysis does not reveal outliers and the variances are low for all the particle size range.

Standardization procedure

Vote on ISO/CD 21083-1 (draft method for 20 – 500 nm)
"Do you agree to the circulation of the draft as a DIS?"
Date of circulation: 2016-06-30
Vote due date: 2016-08-31
Vote results: 11x yes, 3x yes with comments, 1x no, 2x abstain (Attachment 3)
A large amount of comments were received, discussed in TC 195 WG6 meeting in Atlanta, Sept 17 2016, and will be addressed in the next version of the draft.

Standardization procedure

Vote on ISO/CD 21083-2 (draft method for 3 – 20 nm)
"Do you agree to the circulation of the draft as a DIS?"
Date of circulation: 2016-07-01
Vote due date: 2016-08-31
Vote results: 11x yes, 3x yes with comments, 1x no, 2x abstain (Attachment 4)
A large amount of comments were received, discussed in TC 195 WG6 meeting in Atlanta, Sept 17 2016, and will be addressed in the next version of the draft.

Summary

- Standard development for airborne nanoparticle filtration in the range of 3 – 500 nm is underway.

- Round-robin tests are close to the end.

- Statistical analysis of the test data is underway. The repeatability and reproducibility depend on the filter media properties.

- Future activities:

  Further analysis of the test results;

  Revision and improvement of the test methods;

  Circulation of the test methods and development of consensus documents

Thank you

Test procedure (1/2)

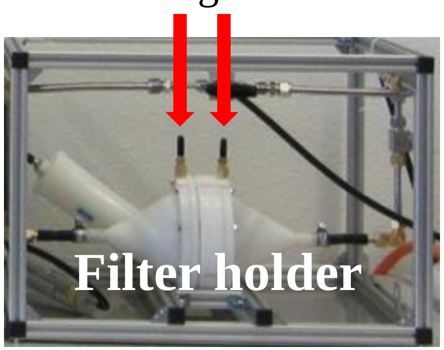
1. Preparatory checks

- The accuracy of instruments should be with in the specification of the manufacturers.
- Zero check, purity check for test air and leakage check should be performed.

2. Pressure drop measurement

- Initial air pressure drop of clean filters should be measured.

Measuring location



3. Correction factors

- The factors, considering particles loss caused by the filter holder and filter supporting screen, should be determined before the filtration tests.

Test procedure (2/2)

4. Measurement of the efficiency of the filters

Particle	Measuring points (Suggested points)		Concentrations
Silver (3 - 20 nm)	6 point (3, 5, 8, 10, 15, 20 nm)		-
	8 points (3, 5, 8, 10, 12, 15, 18, 20 nm)		-
DEHS (20 - 500 nm)	9 points (Tested)	20, 30, 45, 67, 100 and 150 nm	0.03%, diluted in IPA
		224, 335 and 500 nm	0.3%, diluted in IPA
	12 points	20, 25, 30, 41, 56, 77, 105 and 143 nm	0.03%, diluted in IPA
		196, 268, 366 and 500 nm	0.3%, diluted in IPA

5. Minimal downstream counts

Particle size range (nm)	Minimal downstream counts
3 - 50	10
50 - 500	20

6. Test evaluation

Filter grade	Minimal number of testing samples
Low grade	5
High grade	3