Calibration of PN PTI instruments

Vert Forum 2022
Online Event, March, 24th, 2022

Michael Heuser, Oliver Franken

Basic Challenges of PN PTI Instrument Calibrations

Meeting PTI Market Needs Without Cutting too much Corner

Tradeoff between Measurement Uncertainty and Market Needs

- Calibration of PN counters for vehicle type approval in aerosol laboratories can easily cost a four digit number (Euro).
  - Price is a result of expensive (150000+ Euro) reference instrumentation, larger scope of checks and high skill level of reference equipment operators.
  - For PTI instrumentation this is not acceptable.
  - Simplified calibration processes meeting the cost targets of the PTI market are needed.
  - Preferably the subsequent calibration/verification of PN PTI counters can be conducted on site in the workshop or test lanes.
    - Portable, inexpensive and accurate reference instruments needed.

- Challenge: Meeting cost requirements while safeguarding high quality measurements.
  - Avoidance of false passes and false failures are key to public acceptance of the new measurements process during PTI due to high cost implications for vehicle owners in case of a failed PN PTI test.

Challenge: In Field Verification/Calibration

Large markets like Germany with an envisaged quantity of 30000+ PN PTI analyzers in the field often require calibration solutions that are usable on site.

- Consequences:
  - Much larger population of reference instrumentation required compared with the number of fully equipped and accredited aerosol laboratories.
  - Inexpensive solutions required to meet cost constraints

Challenge: Availability of Measurand for Calibration

- Unlike for the calibration of a gas analyzer utilizing calibration gas bottles with known concentrations of certain specimen there are no suitable particles for calibrating PN counters available in bottles.
  - The aerosol needs to be produced in situ where the calibration is conducted.
  - The aerosol needs to have certain properties, mainly characterized by:
    - Particle size distribution, defined by Geometric Standard Deviation (GSD) and Geometric Mean Diameter (GMD)
    - By definition, particles to be used for the calibration need to be **solid**.
    - Certain concentration levels need to be applied.

Overview of Requirements for Reference Instrumentation in Countries implementing PN PTI

Metrological Requirements for Reference Counters, Particle Generators and Devices Under Test

PN PTI Implementing Countries Addressing Calibration Needs

- Currently, four European countries have announced to implement PN PTI: The Netherlands (starting July, 1st 2022), Belgium (starting July, 1st 2022), Switzerland (starting January, 1st 2023) and Germany (starting January, 1st 2023).
- PN PTI activities also in several EU member states and UK.
- European commission is currently looking into establishing an EU guideline for PN PTI
  - Proposal of EU´s Joint Research Centre (JRC) is under review of expert groups
- As of now, Germany, Switzerland and The Netherlands have different instrument requirements as well as different requirements for (subsequent) verification of PN PTI instruments. Belgium is implementing identical requirements as The Netherlands.
- In Switzerland subsequent verification is conducted directly at Metas (Swiss Metrology Institute) due to a comparably small envisaged instrument population. Subject to national measurement ordinance (“Eichrecht”).

Metrological Key Requirements for Subsequent Verification of PN PTI Instruments in Selected Countries – Test Aerosol

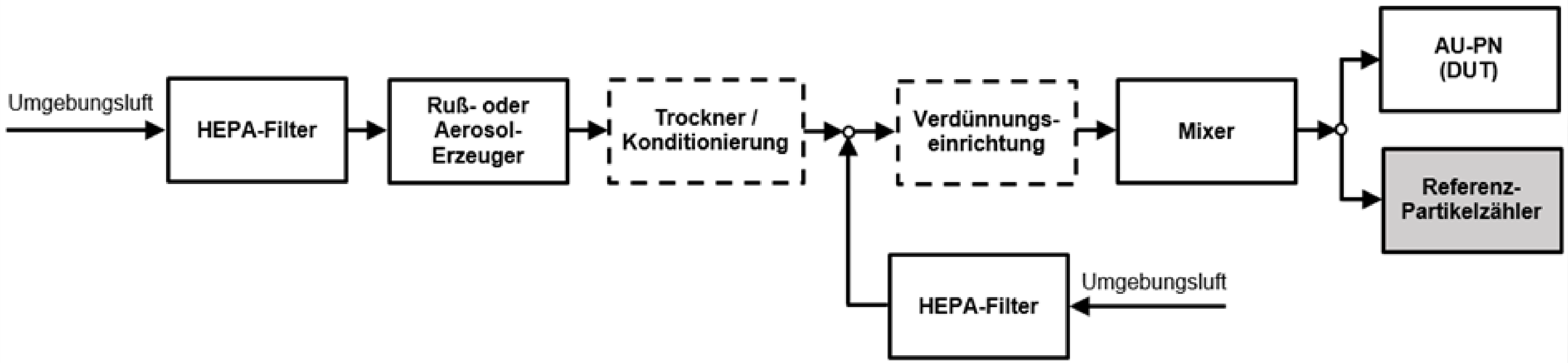
		DE	NL + BE (NMi suggestions)	Comment
	Calibration/Verification Interval	1/a	1/a	
Test Aerosol Requirements	**GMD**	70 nm +- 20 nm	not explicitly defined for subsequent verification	NL + BE: NMi recommendation could be interpreted as polydisperse aerosol w. 80 nm GMD
	GSD	1.5-2.1 (polydiserse)	not explicitly defined for subsequent verification	
	Nominal Concentrations	minimum 3 concentrations:	minimum 3 concentrations:	
		50000 #/cm^3 250000 #/cm^3 300000 - 500000 #/cm^3	"near zero" 100000 #/cm^3 1000000 #/cm^3	
	Tolerance for nominal concentrations	+-20%	+-50%	
	Particle Source	"e.g. salt, soot", also vehicle exhaust is not excluded, but not realistic (uncontrolled characteristics)	"The particles should be close to soot. Other particles (such as salt or oil) may be used provided that correlation is available."	

Metrological Key Requirements for Subsequent Verification of PN PTI Instruments in Selected Countries – Reference PN Counter and DUT

		DE	NL + BE (NMi suggestions)	Comment
Reference Counter Requirements	**State**	solid, thermally stable	solid, thermally stable	
	Measurement Uncertainty	+-50% (expanded measurement uncertainty)	1/3 of MPE (expanded measurement uncertainty), i.e. 8.33%	NL + BE: Practically this could only be achieved by a direct calibration of the reference to the normal of a National Metrology Institute
	Maximum Permissible Error (MPE) for DUT	+-75 % or 10000 #/cm^3 whichever is greater	+-25% or 25000 #/cm^3 whichever is greater	DE: The very large MPE is a result of a long calibration chain and subject to future revisions based on field experience. MPE expected to decrease in the future.

Note: All countries mandate the usage of a reference counter.

Example Calibration Setup (German Calibration Guideline)



Umgebungsluft	Ambient air
HEPA-Filter	HEPA filter
Ruß- oder Aerosol-Erzeuger	Aerosol generator
Trockner / Konditionierung	Dryer/conditioning
Verdünnungseinrichtung	Dilution device
Mixer	Mixer
AU-PN (DUT)	PN PTI unit (DUT)
Referenz-Partikelzahler	Reference particulate counter

Feasibility Considerations

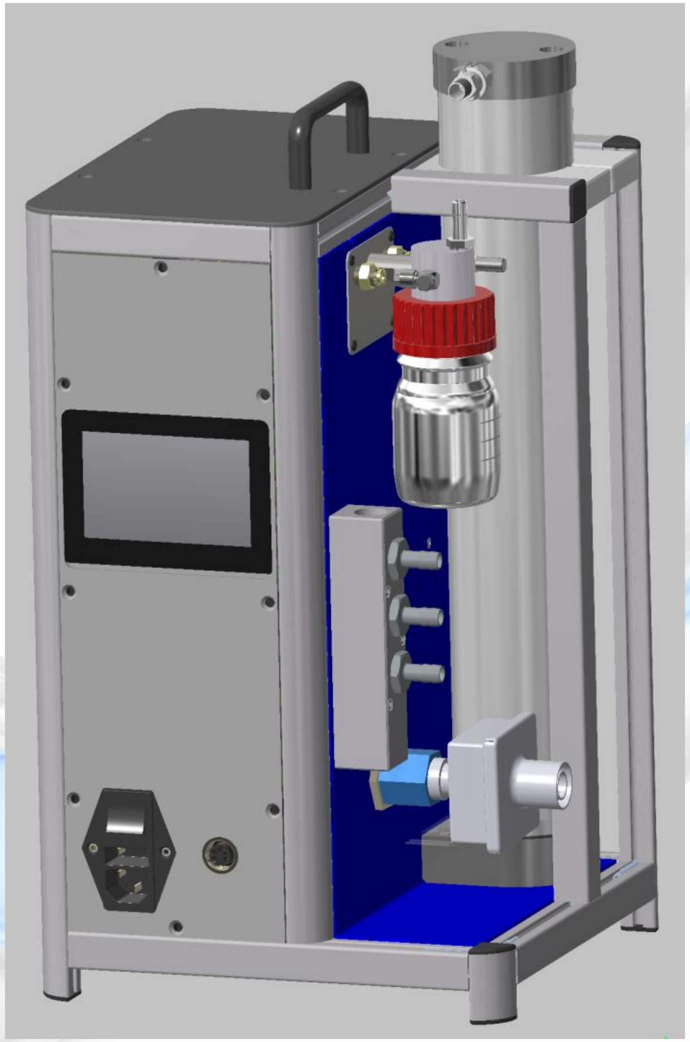
PN PTI related considerations of Aerosol Generators and Reference Counters – Safeguarding High Quality PN PTI Measurements

Options for Aerosol Generation for PN PTI Calibrations

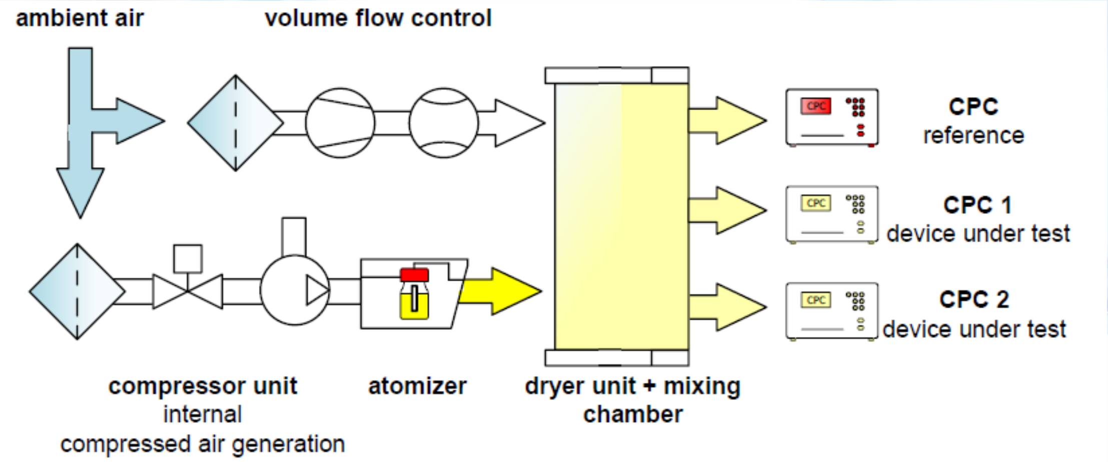
- Most common in aerosol laboratories: Soot Particle Generators
  - Aerosol generation by propane diffusional flame or spark soot generator
  - Advantage: Morphology of generated soot particles is very similar to particles emitted by ICEs, well established in aerosol laboratory and filter test applications
  - Disadvantage: Propane bottle + Nitrogen bottle needed, removal of volatile particles required; for spark soot generators carrier gas bottle needed (Nitrogen or Argon), comparably costly

- Alternative: Salt Particle Generators
  - Aerosol generation by atomizing a salt solution
  - Advantage: PN counters (CPCs) have proven good correlation after calibration on salt particles measuring soot particles and vice versa, usage of medical salt solution contributes to good repeatability of concentrations and size distribution, comparably inexpensive, no gas bottles needed, well established in filter test applications, Combined Particle Generator/Flow Units already commercially available
  - Disadvantage: Generated particles can be pre-charged, potentially problematic for DC based DUTs, particle size distribution can slightly change with different concentration levels, moist aerosol needs to be dried.

Combined Salt Particle Generator/Flow Unit in Detail

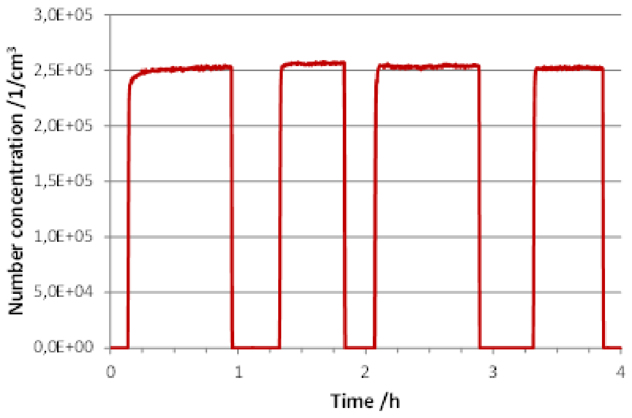


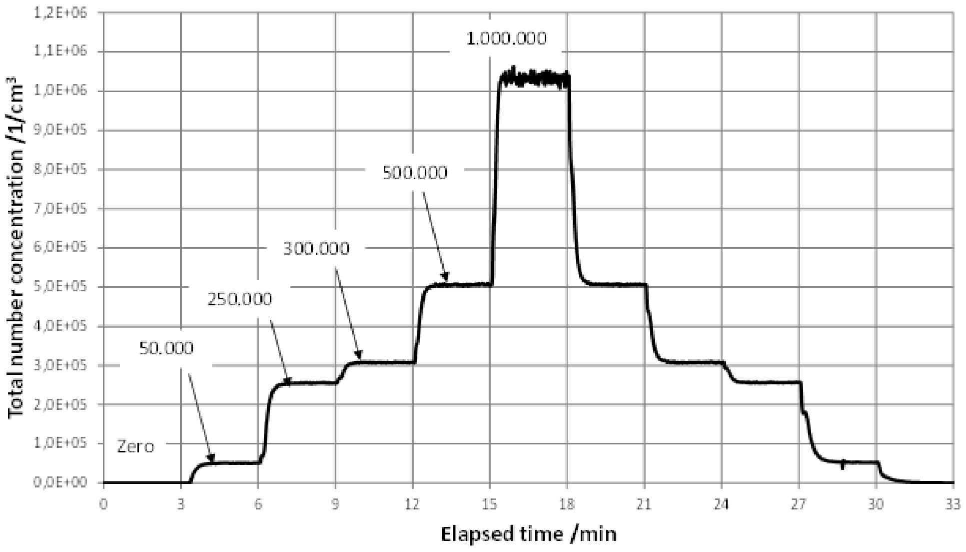
- Polydisperse aerosol (NaCl)
- Monomodale size distribution
- Mean particle size 70 nm +/- 20 nm
- Geometric standard deviation GSD 1,5 – 2,1
- Total number concentration
  - 50k 1/cm³
  - 250k 1/cm³
  - 300k – 500k 1/cm³
  - 1000k 1/cm³



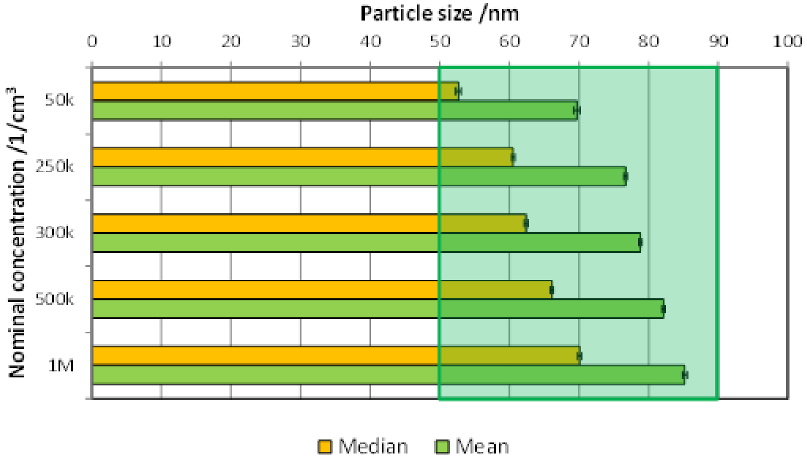
Properties of a Salt Particle Generator

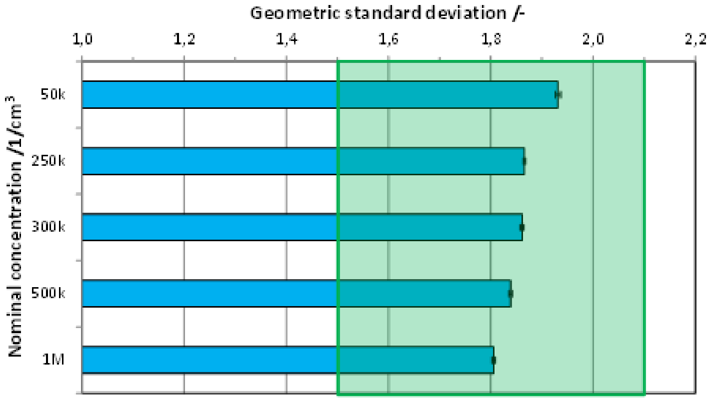
- Concentration repeatablity and steps





- Particle size and standard deviation (70 nm +/- 20 nm, GSD 1,5 – 2,1)



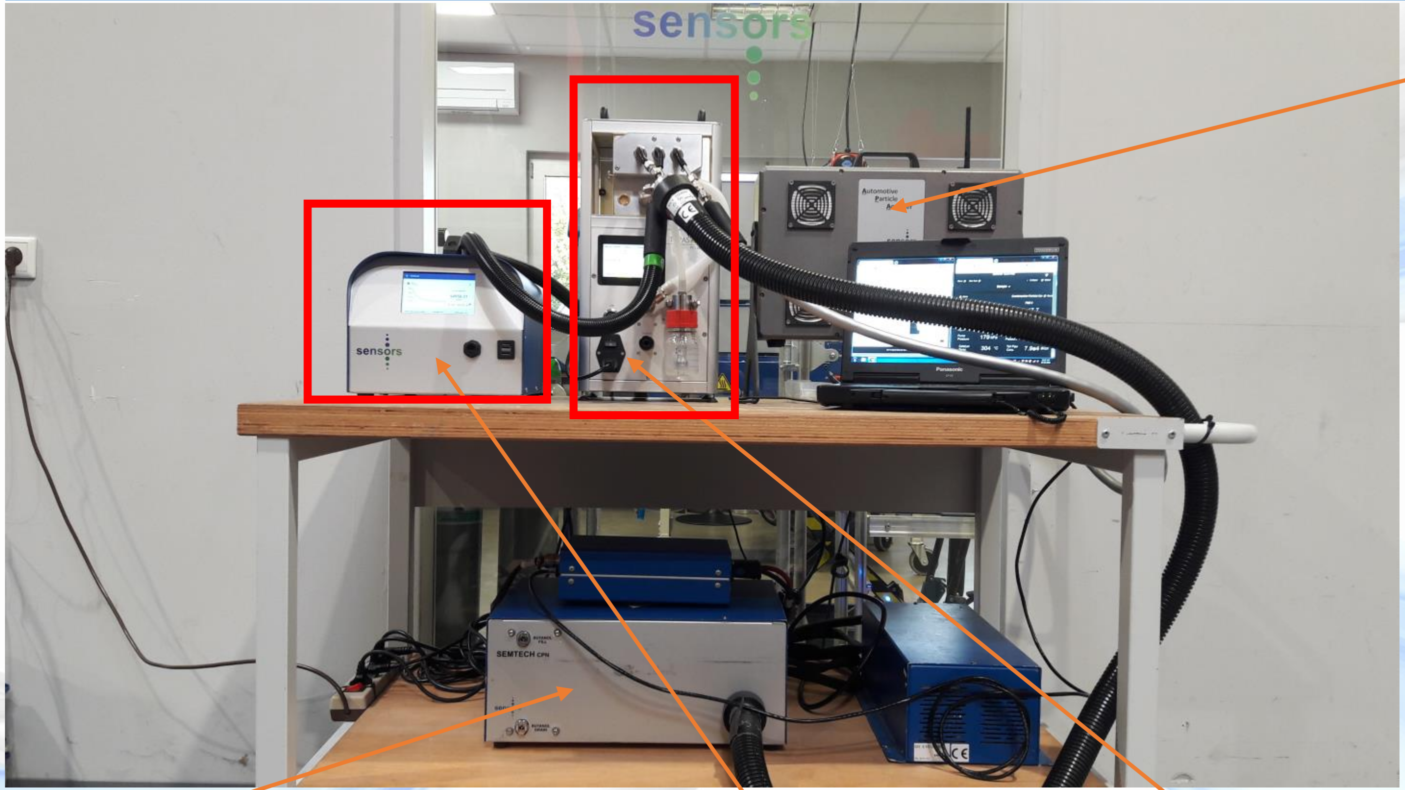


Data source: Topas GmbH

Reference PN Counters – Accuracy Counts

- Reference PN counters need to be traceably calibrated.
- Low measurement uncertainty is key for safeguarding quality measurements of the DUTs in the field.
- Reference PN counters should:
  - Have minimal particle size dependency to cover potentially varying particle size distributions of the test aerosol.
  - Not be biased by pre-charged particles.
  - Always have a much smaller measurement uncertainty than the Device Under Test.
  - Make use of a technology that allows for the application of a well established and most stringent standard – ISO 27891.
  - Be assessed for their measurement uncertainty according to ISO 17025.
  - Meet the cost constraints of the PTI market. Taking cost and complexity out of the Aerosol Generator is not problematic as long as the reference counter can account for potentially increased uncertainties compared with laboratory grade equipment.
  - Ideally use the de facto standard technology for counting PN.
  - For reasons mentioned above use CPC technology.

Evaluated Calibration Setup



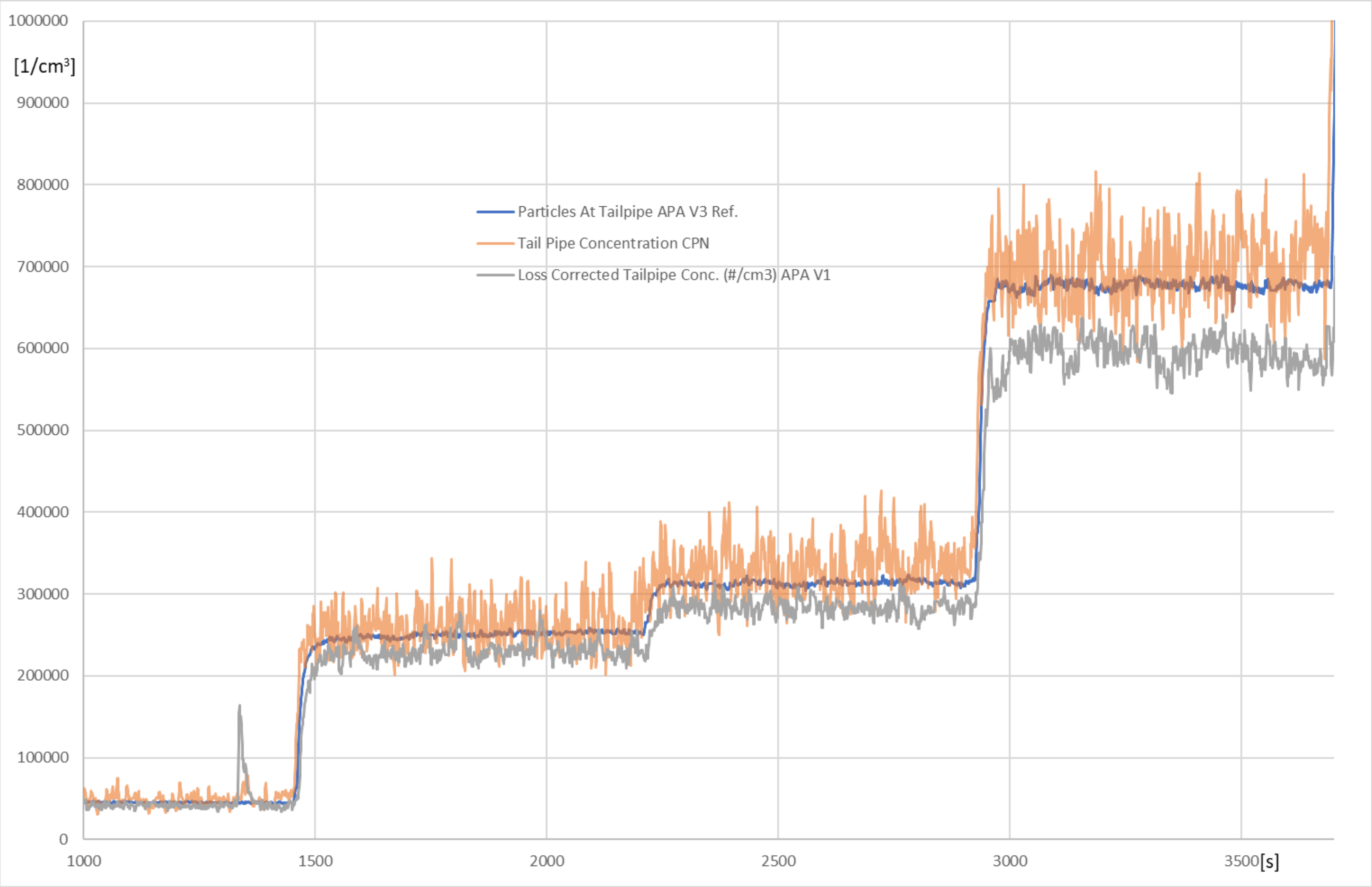
DUT1
Sensors APA
Prototype PN PTI device
(2018), DR=1:100
Sample Flow ~ 1,1 l/min

DUT2
Sensors Semtech CPN
PN PEMS
DR=1:1200
Sample Flow ~ 3,5 l/min

Sensors APA (Reference)
Prototype (2020)
DR=1:10
Sample Flow ~1,3 l/min

Topas FCS 249 Salt Particle Generator/Flow Unit
Prototype

Concentration Traces



Reference and DUT readings over time;
Preadjusted Concentration Settings:
50000 1/cm^3
250000 1/cm^3
300000 1/cm^3
500000 1/cm^3
1000000 1/cm^3

Sensors PN Reference Counter for PN PTI Instrument Calibration

- Turnkey PN reference counter includes:
  - A mixing type Condensation Particle Counter (CPC) that can be calibrated to meet ISO 27891 requirements.
  - Single stage dilution.
  - Full sample conditioning (water removal, thermal sample conditioning) for usage with a wide variety of particle generators.
  - A display for concentration readings and warnings.
  - Safe and convenient means of handling working fluid incl. safety measures for transport and shipment.
  - An interface capable of streaming 1 Hz data to third party calibration software tools.

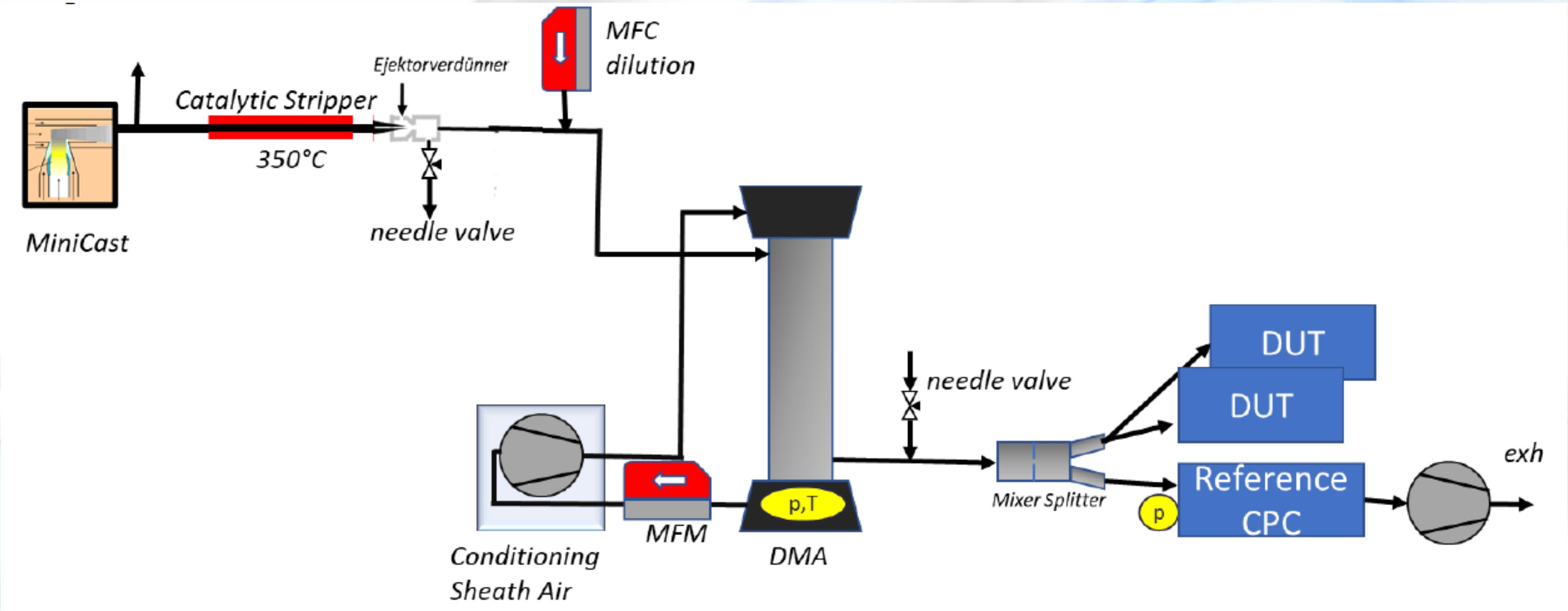
Photo shows prototype

Lab Performance Data

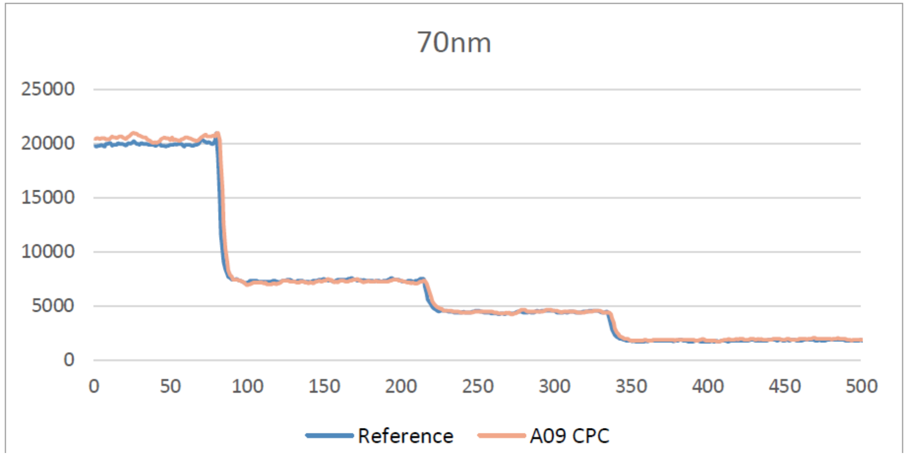
Generated with a Sensors PN PTI Pre Series Device (APA) in a Fully Equipped Aerosol Laboratory

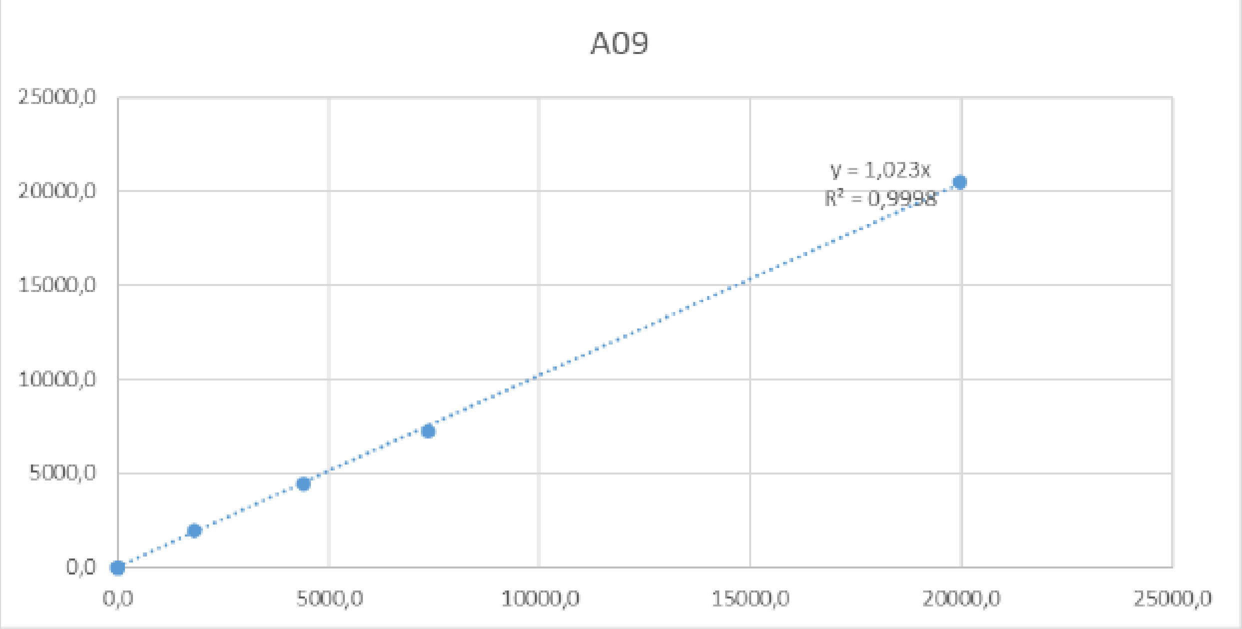
Sensors APA – Performance in Aerosol Lab.

Typical Aerosol Lab. Test Setup



CPC Linearity

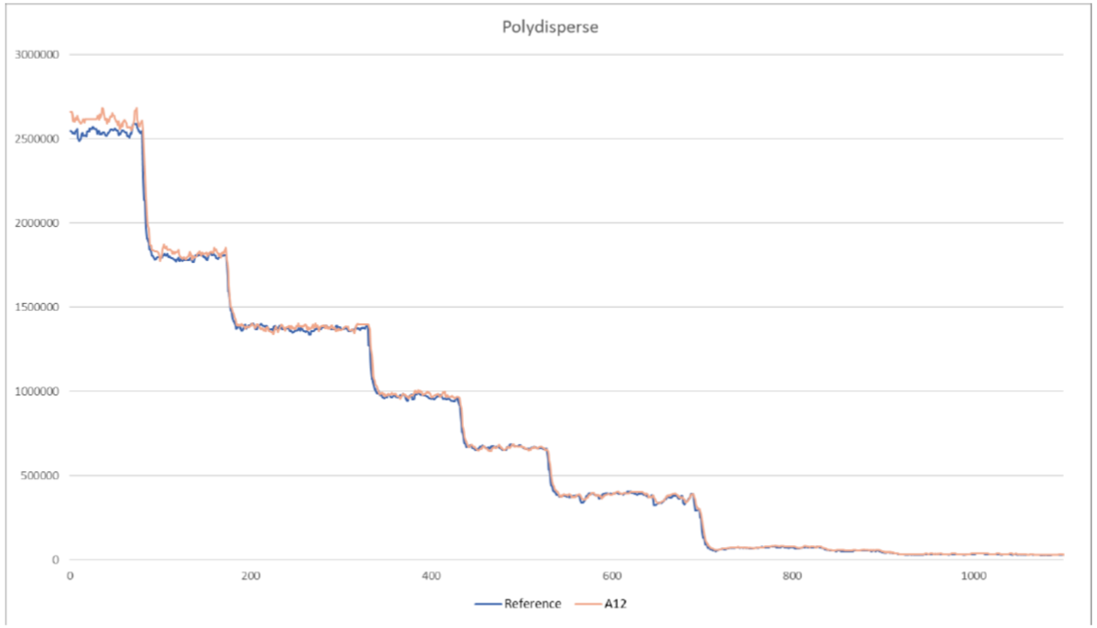




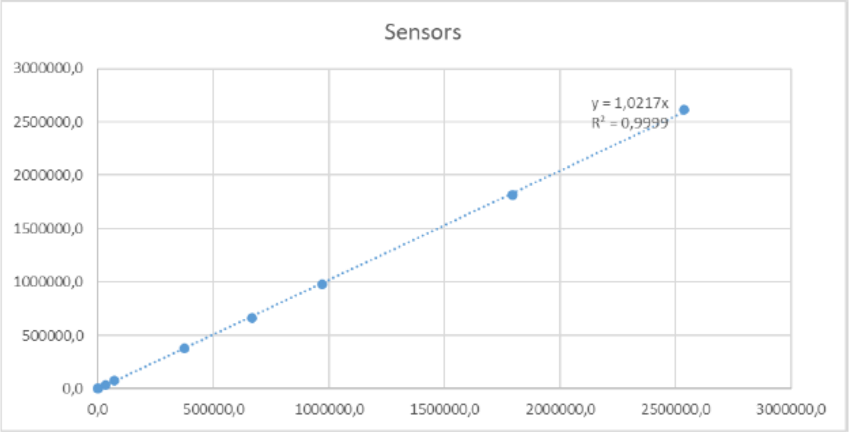
Ref [#/cm³]	A09 CPC [#/cm³]	A09 CPC [%]
19957,6	20521,9	102,8%
7354,7	7258,7	98,7%
4407,5	4459,6	101,2%
1831,3	1920,9	104,9%

Sensors APA Performance Data (Lab. Scale-MT)

7) Polydisperse check of device A12 with AVL-APC reference



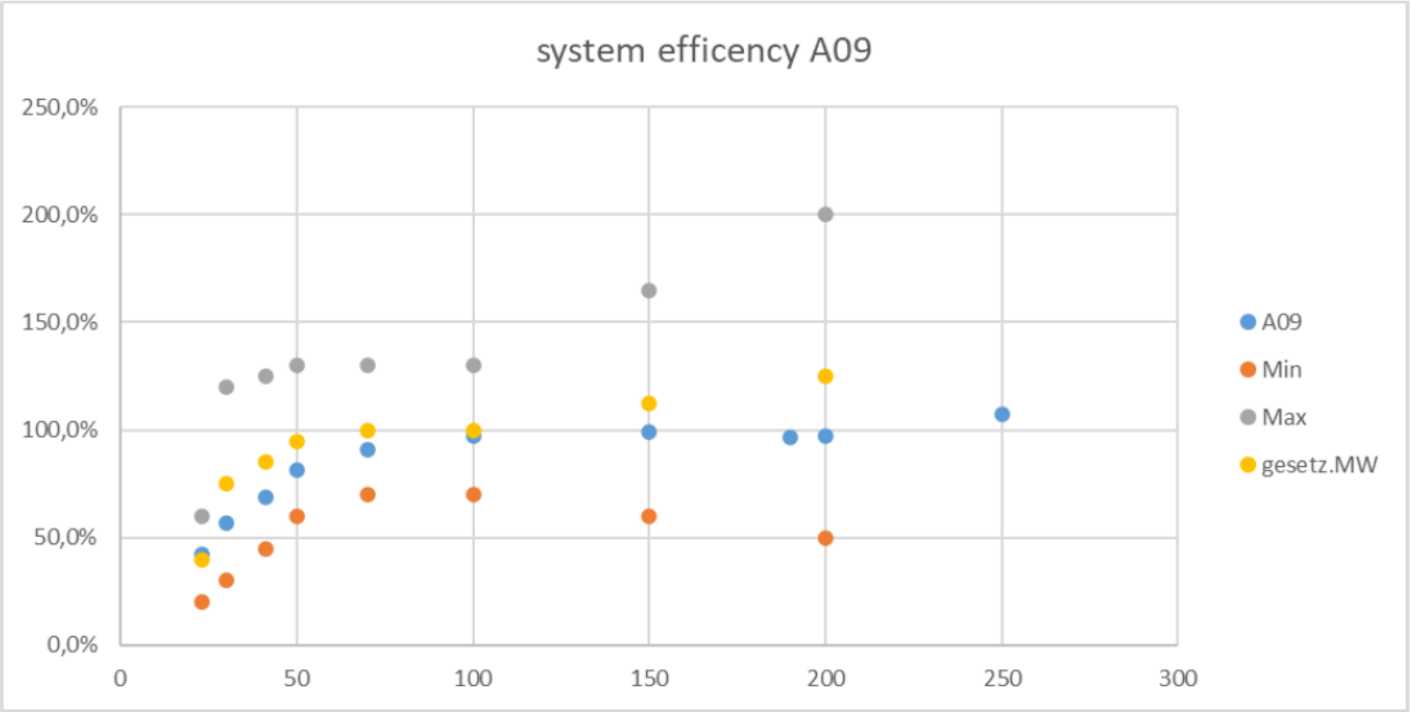
Ref [#/cm³]	A12 [#/cm³]	A12 [%]
2536641,2	2610408,6	102,9%
1795529,8	1820169,4	101,4%
969246,5	981481,4	101,3%
667998,0	664159,0	99,4%
374921,3	381177,0	101,7%
72802,6	76666,9	105,3%
32582,9	34766,8	106,7%



Excellent agreement with lab grade reference equipment on both, polydisperse and monodisperse aerosol (monodisperse data not shown).

Sensors APA Performance Data (Lab. Scale-MT)

Dp	Ref	A09	Dp	A09	gesetzl. Vorgaben		
[nm]	[#/cm³]	[#/cm³]	[nm]	[%]	Min	Max	Mittelwert
23	12935,6	5421,2	23	41,9%	20%	60%	40%
30	8551,8	4851,5	30	56,7%	30%	120%	75%
41	12313,6	8501,8	41	69,0%	45%	125%	85%
50	9339,6	7594,4	50	81,3%	60%	130%	95%
70	13111,7	11913,1	70	90,9%	70%	130%	100%
100	10976,3	10642,9	100	97,0%	70%	130%	100%
150	8673,9	8586,2	150	99,0%	60%	165%	113%
190	7563,7	7298,5	190	96,5%			
200	6153,7	5972,6	200	97,1%	50%	200%	125%
250	2158,4	2313,2	250	107,2%			



Sensors - Worldwide

For further information please contact:

Sensors Europe GmbH, Germany:

- Oliver Franken, oliver.franken@sensors-europe.eu, +49 2104 14188-88
- Michael Heuser, michael.heuser@sensors-europe.eu, +49 2104 14188-75

Sensors, Inc, USA:

- Rob Wilson, rwilson@sensors-inc.com, +1 734 295 9695

Thank you for your attention!