The Traceable Calibration of Condensation Particle Counters

Hans-Georg Horn, Oliver F. Bischof, Axel Zerrath, TSI GmbH, Aachen, Germany 10th ETH-Conference on Combustion Generated Nanoparticles

Introduction

Within the GRPE Particle Measurement Programme (PMP), a Condensation Particle Counter (CPC) is the prime candidate to be used to measure the particle number concentration of emissions during drive cycles for type approval purposes (Liu et al. 2005). Therefore, it is essential to calibrate CPCs using a traceable and independent method to ensure proper performance of the measurement system. Yet unlike for gaseous compounds, no well defined calibration standards exist for particle number concentration. Former presentations and papers (Liu et al. 2004 and Liu et al. 2005) described the method suggested and used in-house by the CPC manufacturer (TSI Inc., St. Paul, MN). The traceability of this calibration method depends on

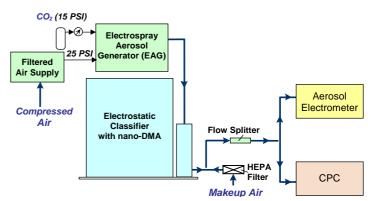


Fig. 1: Flow schematic of the CPC calibration system

- the ability of a nano-DMA to produce singly-charged, monodisperse particles of a known size,
- the ability of an aerosol electrometer to serve as the particle number concentration reference detector.

Calibration Method

Figure 1 shows the flow schematic and a photograph of the calibration system. For the determination of the counting efficiency curve of a CPC, oil droplets are generated by means of a stable, high-concentration electrospray aerosol generator (EAG). This primary aerosol has a sharp peak at the particle size of interest. If

Tab. 1: Traceability of the electrical mobility of a nano-DMA with recirculated sheath flow.

$Z_{p} = \frac{q_{s}}{2\pi VL} \ln \left(\frac{r_{2}}{r_{1}} \right)$					
	Description	Traceable Measurement			
q_s	Recirculated sheath flow	NIST traceable flowmeter			
L	Length between DMA inlet and outlet slit	NIST traceable caliper			
r_{l}	Outer radius of inner electrode	NIST traceable micrometer			
r_2	Inner radius of outer electrode	NIST traceable bore gage			
V	Voltage on the center electrode	NIST traceable kV divider			

Verieble	Uncertainty in variable (%)	Resulting uncertainty in diameter (%)			
Variable		16 nm	18 nm	23 nm	37 nm
L	0.69 ^a	0.35	0.35	0.35	0.36
r_1	0.07 ^b	0.05	0.05	0.05	0.05
r_2	0.07 ^c	0.05	0.05	0.05	0.05
q_s	2 ^d	1	1.01	1.01	1.03
V	1.41 ^e	0.71	0.72	0.72	0.74
Temperature	0.34 ^d	0.05	0.05	0.05	0.05
Pressure	1 ^d	0.16	0.17	0.17	0.19
Slip correction factor	0.9 ^f	0.61	0.61	0.61	0.61
Combined uncertainty in diameter	-	1.43	1.44	1.44	1.47

^{a.} From nano-DMA length analysis

^{b.} From manufacturer's tolerance design drawing

^{c.} From manufacturer's tolerance design drawing

d. From TSI 4100 flowmeter specification

^{e.} Combined uncertainty of the Hallmark Standards voltage divider (1 %) and the Fluke 45 Dual Display Multimeter (1%)

^{f.} Based on 100 nm particles, estimated by NIST (Kinney et al., 1991)

classified with a nano differential mobility analyzer (nano-DMA), the result is a highly monodisperse (geometric standard deviation < 1.05), singly-charged calibration aerosol of known size. The calibration aerosol is split equally between a reference aerosol electrometer and the CPC(s) to be calibrated. Apart from the particle size dependent counting efficiency, also the concentration linearity response can be tested with

this setup. Larger particles ($D_p = 50$ nm) are typically used for this test to ensure 100% counting efficiency of the CPC. A dilution bridge between the EAG and the nano-DMA is used to provide a number of different concentration levels between 2,000 and 10,000 cm⁻³.

Traceability and uncertainty of the calibration

For a nano-DMA with recirculated sheath flow, the electrical mobility (Z_p) of the selected particles is defined according to the equation in Table 1. This table also lists the traceable measurement method used to verify all parameters.

An uncertainty analysis was performed for the nano-DMA similar to the one that was previously done for the long DMA by Kinney et al. (1991). The analysis was based on operating conditions of 23°C and 101.3 kPa. The results are summarized in Table 2. The particle number concentration (*N*) as measured with an aerosol electrometer is calculated based on the particle charge $(e \cdot n_p)$, the sample flow rate (q_e) , and the resistance of the embedded high-gain amplifier (*R*) that converts current into voltage (*V*):

$$N = \frac{V}{e \cdot n_p \cdot q_e \cdot R}$$

The calibration particles are verified to be singly-charged ($n_p=1$) in the size range of interest using an SMPS system. The sample flow rate into the aerosol electrometer is measured with a built-in NIST traceable flow meter ($\pm 2\%$). The embedded resistor was measured by the manufacturer using a NIST traceable standard to ensure its resistance was within $\pm 1\%$ of the nominal value. The electrometer's current measurement circuitry is further calibrated by applying several known currents that are generated by a NIST traceable DC voltage divider and a resistor assembly which is calibrated periodically against a NIST traceable standard by a local calibration test house.

The calibration setup (see Figure 1) includes a symmetrical flow splitter. The tubing from the flow splitter to the CPC(s) and to the reference electrometer is of equal length and all flow rates are identical. This ensures identical particle transport losses (from the flow splitter to the instrument inlet) for the CPC(s) and the electrometer. However, particle losses inside the aerosol electrometer must be considered. Calculating of the internal diffusion losses for the TSI Aerosol Electrometer 3068B shows that for a calibration according to PMP requirements, particle losses in the electrometer are smaller than 1 % for all required particle sizes.

Results

The CPC 3010D has been in production for more than one year. Figure 2 present the results from a subset of the production of 3010D CPCs. The average particle diameter with 50% counting efficiency (D_{50}) is 22.7 nm. For all CPCs, D_{50} is within the PMP range of 23 ± 3 nm. The concentration linearity slope in Figure 2 is between 0.9508 and 1.02, which is within ±5 % of the electrometer reading. The correlation coefficients R² are larger than 0.997. Results indicate the excellent repeat-ability of the counting efficiency and linearity response performance of the CPCs and demonstrate the validity of the calibration method.

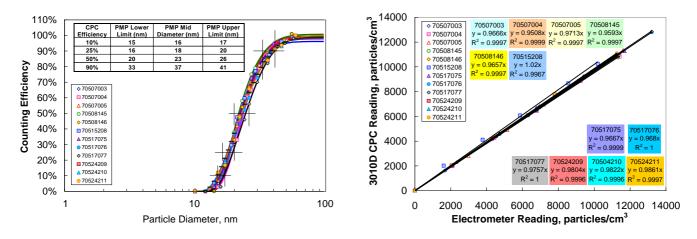


Fig. 2: Counting efficiency results and linearity response for twelve production CPCs 3010D.

References

Liu, W., Osmondson, B.L. Sem, G.J., Andersson, J. and Clarke, D. 2005. UN-GRPE PMP Phase 3 - Interlaboratory Correlation Exercise: Updated. Framework and Laboratory Guide for HD Engine Testing. A Document For The UK Department for Transport. Q55022. May 2005

Liu, W., Osmondson, B.L., Bischof, O.F. and Sem. G.J. 2004. Traceable Calibration of Condensation Particle Counters with Respect to Smallest Particle Size Detection Limit, Counting Efficiency, and Concentration Linearity. Presentation at 8th ETH -Conference on Combustion Generated Nanoparticles, Zurich, Switzerland, August 16-18, 2004.

Kinney, P.D., Pui, D.Y.H., Mulholland, G.W., Bryner, N.P. 1991. Use of the Electrostatic Classification Method to Size 0.1 µm SRM Particles - A Feasibility Study, Journal of Research of the National Institute of Standards and Technology, 96(2):147-176, 1991.

Liu, W., Osmondson, B.L., Bischof, O.F. and Sem, G.J 2005. Calibration of Condensation Particle Counters; SAE Paper No. 2005-01-0189; Emissions Measurement and Testing; Society of Automotive Engineers (SAE): Warrendale, PA, Book No. SP-1941, 2005. SAE 2005 World Congress Detroit, MI, USA, 2005.



The Traceable Calibration of **Condensation Particle Counters**

Hans-Georg Horn, Oliver F. Bischof, Axel Zerrath TSI GmbH, Neuköllner Straße 4, D-52068 Aachen

Introduction

Within the GRPE Particle Measurement Programme (PMP), a Condensation Particle Counter (CPC) is the prime candidate to be used to measure the particle number concentration of emissions during drive cycles for type approval purposes (Liu et al. 2005). Therefore, it is essential to calibrate CPCs using a traceable and independent method to ensure proper performance of the measurement system. Yet unlike for gaseous compounds, no well defined calibration standards exist for particle number concentration.

Basics

Former presentations and papers (Liu et al. 2004 and Liu et al. 2005) described the method suggested and used in-house by the CPC manufacturer (TSI Inc., St. Paul, MN).

The traceability of this calibration method depends on

- the ability of a nano-DMA to produce singlycharged, monodisperse particles of a known size.
- the ability of an aerosol electrometer to serve as the particle number concentration reference detector

Calibration Method

Figure 1 shows the flow schematic and a photograph of the calibration system. For the determination of the counting efficiency curve of a CPC, oil droplets are generated by means of a stable, high-concen-tration electrospray aerosol generator (EAG). This primary aerosol has a sharp peak at the particle size of interest. If classified with a nano differential mobility analyzer (nano-DMA), the result is a highly monodisperse, singly-charged calibration aerosol of known size, see Figure 2. The calibration aerosol is split equally between a reference aerosol electrometer and the CPC(s) to be calibrated.

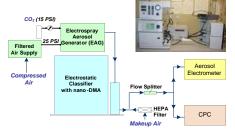


Fig. 1: Flow schematic of the CPC calibration System

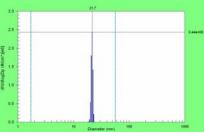


Fig. 2: EAG-generated, DMA-classified monodisperse particles with a peak size of 22 nm and a geometric standard deviation of 1.04

Apart from the particle size dependent counting efficiency, also the concentration linearity response can be tested with this setup. Larger particles (Dp = 50 nm) are typically used for this test to ensure 100% counting efficiency of the CPC. A dilution bridge between the EAG and the nano-DMA is used to provide a number of different concentration levels between 2,000 and 10,000 cm⁻³.

Traceabilty and uncertainty of particle size

For a nano-DMA with recirculated sheath flow, the electrical mobility (Z_p) of the selected particles is defined according to the equation in Table 1. This table also lists the traceable measurement method used to verify all parameters.

Tab. 1: Traceability of the electrical mobility of a nano-DMA with recirculated sheath flow.

$Z_{p} = \frac{q_{s}}{2\pi VL} \ln\left(\frac{r_{2}}{r_{1}}\right)$							
	Description	Traceable Measurement					
q_s	Recirculated sheath flow	NIST traceable flowmeter					
	Length between DMA inlet and outlet slit	NIST traceable caliper					
r_l	Outer radius of inner electrode	NIST traceable micrometer					
r_2	Inner radius of outer electrode	NIST traceable bore gage					
V	Voltage on the center electrode	NIST traceable kV divider					

An uncertainty analysis was performed for the nano-DMA similar to the one that was previously done for the long DMA by Kinney et al. (1991). The analysis was based on operating conditions of 23°C and 101.3 kPa. The results are summarized in Table 2

Tab. 2: Summary of nano-DMA uncertainty analysis

	Uncertainty	Resulting uncertainty in diameter			
Variable	in variable	(%)			
	(%)	16 nm	18 nm	23 nm	37 nm
L	0.69 ^a	0.35	0.35	0.35	0.36
r_I	0.07 ^b	0.05	0.05	0.05	0.05
r_2	0.07 ^c	0.05	0.05	0.05	0.05
q_s	2 ^d	1	1.01	1.01	1.03
V	1.41 ^e	0.71	0.72	0.72	0.74
Temperature	0.34 ^d	0.05	0.05	0.05	0.05
Pressure	1 ^d	0.16	0.17	0.17	0.19
Slip					
correction	0.9 ^f	0.61	0.61	0.61	0.61
factor					
Combined					
uncertainty	-	1.43	1.44	1.44	1.47
in diameter					

- ^{a.} From nano-DMA length analysis ^{b.} From manufacturer's tolerance design drawing
- From manufacturer's tolerance design drawing
- ^{d.} From TSI 4100 flowmeter specification e. Combined uncertainty of the Hallmark Standards
- voltage divider (1 %) and the Fluke 45 Dual Display Multimeter (1%)
- Based on 100 nm particles, estimated by NIST (Kinney et al., 1991)

Traceabilty and uncertainty of the number concentration

The particle number concentration (N) as measured with an aerosol electrometer is calculated based on the particle charge (en_p) , the sample flow rate (q_e) , and the resistance of the embedded high-gain amplifier (R) that converts current into voltage (V):

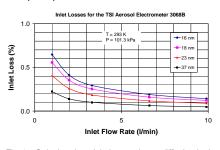
$$N = \frac{V}{e \cdot n_p \cdot q_e \cdot R}$$

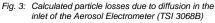
The calibration particles are verified to be singlycharged $(n_p=1)$ in the size range of interest using an SMPS system, see Figure 2. The sample flow rate into the aerosol electrometer is measured with a built-in NIST traceable flow meter ($\pm 2\%$). The embedded resistor was measured by the manufacturer using a NIST traceable standard to ensure its resistance was within $\pm 1\%$ of the nominal value. The electrometer's current measurement circuitry is further calibrated by applying several known currents that are generated by a NIST traceable DC voltage divider and a resistor assembly which is calibrated periodically against a NIST traceable standard by a local calibration test house.

Particle Transport Losses

The calibration setup (see Figure 1) includes a symmetrical flow splitter. The tubing from the flow splitter to the CPC(s) and to the reference electrometer is of equal length and all flow rates are identical. This ensures identical particle transport losses (from the flow splitter to the instrument inlet) for the CPC(s) and the electrometer. However, particle losses inside the aerosol electrometer must be considered. Figure 3 shows a calculation of the internal diffusion losses for the TSI Aerosol

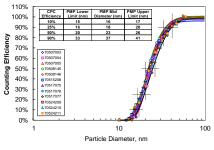
Electrometer 3068B. It is shown that for a calibration according to PMP requirements, particle losses in the electrometer are smaller than 1 % for all required particle sizes.



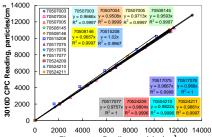


Results

The CPC 3010D has been in production for more than one year. Figure 4 and 5 present the results from a subset of the production of 3010D CPCs. The average particle diameter with 50% counting efficiency (D_{50}) is 22.7 nm. For all CPCs, D_{50} is within the PMP range of 23 ± 3 nm. The concentration linearity slope in Figure 5 is between 0.9508 and 1.02, which is within ± 5 % of the electrometer reading. The correlation coefficients R² are larger than 0.997. Results indicate the excellent repeatability of the counting efficiency and linearity response performance of the CPCs and demonstrate the validity of the calibration method.



4: Counting efficiency results for twelve Fig. production CPCs 3010D



neter Reading, particles/cm

Fig. 5: Linearity response of twelve production CPCs 3010D.

References

Liu, W., Osmondson, B.L. Sem, G.J., Andersson, J. and Clarke, D. 2005. UN-GRPE PMP Phase 3 - Inter-laboratory Correlation Exercise: Updated, Framework and Laboratory Guide for HD Engine Testing. A Document For The UK Department for Transport. Q55022. May 2005

Liu, W., Osmondson, B.L., Bischof, O.F. and Sem. G.J. 2004. Traceable Calibration of Condensation Particle Counters with Respect to Smallest Particle Size Detection Limit, Counting Efficiency, and Concentration Linearity. Presentation at 8th ETH -Conference on Combustion Generated Nanoparticles, Zurich, Switzerland, August 16-18, 2004.

Kinney, P.D., Pui, D.Y.H., Mulholland, G.W., Bryner, N.P. 1991. Use of the Electrostatic Classification Method to Size 0.1 µm SRM Particles - A Feasibility Study, Journal of Research of the National Institute of Standards and Technology, 96(2):147-176,

Liu, W., Osmondson, B.L., Bischof, O.F. and Sem, G.J 2005 Calibration of Condensation Particle Counters; SAE Paper No. 2005-01-0189; Emissions Measurement and Testing; Society of Automotive Engineers (SAE): Warrendale, PA, Book No. SP-1941, 2005. SAE 2005 World Congress Detroit, MI, USA, 2005.