Volatile Particle Remover
Calibration Procedure

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AEA Energy & Environment  
Building 551.11  
Harwell International Business Centre  
Didcot  
OX11 0QJ

t: 0870 190 6566  
f: 0870 190 6608

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AEA Technology plc

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<table>
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<tr>
<th><strong>Author</strong></th>
<th>Name</th>
<th>Emma Sandbach</th>
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<tr>
<td><strong>Approved by</strong></td>
<td>Name</td>
<td>Ian Marshall</td>
</tr>
<tr>
<td><strong>Signature</strong></td>
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# Table of contents

1 Introduction 1
2 VPR Overview 2
3 Calibration Methods 3
   3.1 Calibration Criteria 3
   3.2 Calibration Aerosol 4
   3.3 Aerosol generation and VPR calibration method: example 4
4 Procedure 9
   4.1 Calibration of the VPR for solid particle penetration efficiency 9
   4.2 Calibration of the VPR for volatile particle removal efficiency (supression of nucleation particles) 11
   4.3 Calculation of penetration efficiency of solid particles 11
   4.4 Calculation of supression of nucleation particles 12
5 References 13
1 Introduction

The ‘Conclusions on improving particulate mass measurement procedures and new particle number measurement procedures relative to the requirements of the 05 series of amendments to Regulations No. 83, GRPE-48-11-Rev.1’ and the ‘UN-GRPE PMP Phase 3 Inter-laboratory Correlation Exercise: Framework and Laboratory Guide’ describe a sampling pre-conditioning system to be used for particle number emissions measurements.

The major component of the sample pre-conditioning system is a Volatile Particle Remover (VPR), which is used to define the nature of the particles to be measured.

The VPR provides heated dilution, thermal conditioning of the sample aerosol, further dilution of particle number concentration and cooling of the sample prior to entry into the particle number counter.

This document describes the calibration procedures for the VPR.
2 VPR Overview

The VPR comprises three components; a particle number diluter (PND₁), an evaporation tube (ET) and a second particle number diluter (PND₂); these are illustrated in the red box Figure 1. Each component is described in more detail below (taken from the UN-GRPE PMP Phase 3 Inter-laboratory Correlation Exercise: Framework and Laboratory Guide²).

First Particle Number Diluter (PND₁)
The PND₁ diluter shall be specifically designed to dilute particle number concentration and output a dilute sample equal to 150°C +/- 5°C. The diluter should be supplied with HEPA filtered dilution air and be capable of a dilution ratio range of 1 to 1000 times.

Evaporation Tube (ET)
The ET shall be a length of tubing 240mm +/-10 mm and I.D 6mm +/- 0.1mm equipped with a heating mantle. The entire length of the ET must be controlled to a temperature greater than that of PND₁, with a portion of the length equivalent to a gas residence time of 0.2s +/- 0.05s held at a constant temperature (+/-20°C) of 300°C.

Second Particle Number Diluter (PND₂)
The PND₂ device shall be specifically designed to dilute particle number concentration. The diluter shall be supplied with HEPA filtered dilution air and be capable of a dilution ratio of ~10 times. The dilution ratio of this diluter; (PNDR₂) is selected such that the particle number concentration downstream of the PND₂ diluter is <10⁴ particles/cm³ and the gas temperature prior to entry to the Particle Number Counter (PNC) is <35°C.

Figure 1: Schematic diagram of particle number emissions measurement system
3  Calibration Methods

The following general points are brought to the attention of the reader:

1. Principles of good calibration practice should be adopted when using any of the methods described below. In particular, users should ensure that any piece of ancillary equipment that is critical to the calibration is itself appropriately calibrated before use.

2. The calibration methods rely upon comparing the measurements from two pieces of aerosol measurement instrumentation. The user must ensure that the calibration aerosol pathway to each instrument is configured so that any particle deposition that may occur between particle source and instrument is comparable for both instruments. This is usually achieved by ensuring residence time is comparable in both pathways. It may also be necessary to take into account the pathway within each instrument from the inlet to the measurement volume, if there are significant differences between the instruments.

3. Particle deposition can be minimised by utilizing conductive tubing.

4. The term ‘upstream’ refers to the sampling location at the inlet of the VPR ie PND₁ inlet.

5. The term ‘downstream’ refers to the sampling location at the outlet of the VPR ie after the second diluter PND₂.

3.1  Calibration Criteria

The VPR must be calibrated in terms of its solid particle penetration efficiency and its volatile particle removal efficiency (or suppression of nucleation particles).

3.1.1  Solid particle penetration efficiency

The test aerosol for these measurements shall be solid particles of diameters 30, 50 and 100 nm and a minimum concentration of 1,000 particles cm⁻³. Particle concentrations shall be measured upstream and downstream of the apparatus operating at the temperature and flow conditions employed during an emission test. A minimum penetration efficiency of 80% shall be achieved at all three test particle diameters.
3.1.2 Volatile particle removal efficiency

The VPR shall operate under conditions that achieve greater than 99% reduction of 30nm C40 (tetracontane) particles.

3.2 Calibration Aerosol

3.2.1 Solid particles

There are a number of aerosol generation techniques that are capable of producing solid particles in the size range between 30 and 100nm diameter including condensation generators, combustion aerosol generators, nebulisation and hot wires. Any of these methods are suitable for producing calibration aerosols providing they meet the following criteria:

- Capable of producing a stable aerosol at a minimum concentration of 1,000 particles cm\(^{-3}\) and a maximum concentration of 10,000 particles cm\(^{-3}\).
- Monodispersity: GSD ≤ 1.2

A common aerosol used for instrument calibration is sodium chloride. This is well characterised, inexpensive and relatively safe to use compared with other aerosols. Sodium chloride particles may be generated using a number of the techniques listed above.

3.2.2 Volatile particles

The requirement for volatile particles is more prescriptive than for solid particles: tetracontane (C40) particles with a diameter of 30nm must be produced to assess the volatile particle removal efficiency of the VPR. A condensation generator is a simple method to produce these particles.

A technique for producing solid and volatile particles is described in the example below. Alternative methods that generate similarly sized particles and concentrations are equally appropriate.

3.3 Aerosol generation and VPR calibration method: example

This approach has been adopted to calibrate the VPR within the Golden Particle Measurement System (GPMS) that was used in the light duty PMP Inter Laboratory Comparison Exercise (ILCE).

The experimental set-up is illustrated in Figure 2.
The aerosol generator consisted of a ceramic crucible heated via an electric Bunsen (Figure 3). The bulk material (sodium chloride for solid particle penetration efficiencies and tetracontane for volatile particle removal efficiencies) was placed in the ceramic crucible and heated to near its boiling point. A small flow was introduced into the crucible to displace vapour from the surface of the bulk material to a cooler region of the generator where condensation occurred. Particle diameters were varied by controlling the rate of vapour transport from the crucible (via the crucible air flow) and/or the subsequent cooling rate of the vapour (via the carrier air flow). A typical polydisperse size distribution (before selection of a monodisperse fraction) of a generated aerosol is presented in Figure 4.
Figure 3: Condensation Aerosol Generator

Filtered Carrier Air

Crucible Air Flow

Condensation

Aerosol Outlet

Filtered Carrier Air Aerosol Out

Crucible Air Flow

Heater
Note: This aerosol generation method is reported purely as an example. Alternative methods that generate similarly sized particles and concentrations are equally appropriate.

The electrostatic classifier was used to select monodisperse fractions of the polydisperse calibration aerosol and deliver the required number concentrations to the VPR (> 1000 particles cm\(^{-3}\) for sodium chloride and >10,000 particles cm\(^{-3}\) for tetracontane), reducing the GSD (geometric standard deviation) to around 1.2.

Two CPCs were used to simultaneously record the number concentrations upstream (at the inlet of PND\(_1\)) and downstream (at the outlet of the VPR, ie the PNC\(_{\text{GOLD}}\) position).

Note: It is imperative that the response of these CPCs is verified before the VPR is calibrated; this is to ensure that the two CPCs are equivalent. The upstream and downstream concentrations are directly compared with each other to calculate the penetration efficiencies of the VPR; therefore the CPCs must be compared with each other. The secondary calibration method for CPC calibration is recommended, as detailed in the ‘Condensation Particle Counter Calibration Procedures\(^3\). The linearity of response in terms of the \(R^2\) coefficient must be greater than 0.98, else the CPCs do not respond linearly over the measured concentration range. The gradient of the calibration plot must be within the range from 0.95 to 1.05 (unity signifies complete agreement between the two CPCs). If the two CPCs do not meet these requirements the upstream and downstream concentrations may not be directly compared to calculate the penetration efficiencies. The results from the CPCs would then need to be corrected to take into consideration any measurement differences between the two.
unfavourable as this introduces additional measurement uncertainties into the calibration procedure of the VPR.

Additionally, care must be taken when comparing the results from two CPCs if they are not the same model, this was particularly important in this example as PNC_GOLD had been modified to provide lower size detection limits of 23±3 and 37±4 nm for 50 % detection efficiency (D_{50}) and 90 % detection efficiency (D_{90}) respectively than the standard model of TSI CPC 3010. The AEA CPC was unmodified having a D_{50} of 9.5±0.5 nm and D_{90} of 13.5±0.5 nm. Thus significant differences between the concentrations reported by both CPCs would be expected if the calibration particles were smaller than around 40 nm. For the VPR calibrations at 30nm the AEA CPC was used in both the upstream and downstream sampling positions through equivalent sampling lines. The downstream sampling line was exchanged for the upstream sampling line, the number concentration was allowed to stabilise and then the upstream sampling was reconnected to the CPC to verify that the inlet concentration had not drifted throughout the measurements.

Identical flow paths were used to deliver the calibration particles from the electrostatic classifier to the AEA CPC, PNC_GOLD and the VPR inlet, thus ensuring any losses within the pipe work were the equivalent for all measurements.

For each monodisperse diameter the system was allowed to stabilise for at least 10 minutes before selecting the next diameter. The VPR was calibrated at the dilution settings most commonly used in the ILCE.

When the measurements at each diameter had been made the results were analysed to calculate the penetration efficiency of solid particles and supression of nucleation particles.
4 Procedure

The procedures for performing a VPR calibration are described below for each type of particle. It is assumed that the correlation between the two CPCs (at the inlet and outlet of the VPR), has been confirmed before this procedure commences, using the secondary calibration method described in the ‘Condensation Particle Counter Calibration Procedures’ document.

Note: *PNC_REF measured particle concentrations are not required for this calibration, it is simply operated to maintain the appropriate flow rates within the VPR.*

4.1 Calibration of the VPR for solid particle penetration efficiency

Prepare the VPR and CPCs for use:

- Position all apparatus as described in Figure 2.
- Where appropriate clean any dilution mechanisms within the VPR as advised by the manufacturer (eg for rotating disc diluters remove the disc and clean using an appropriate solvent).
- Perform any routine maintenance of the VPR as advised by the manufacturer (eg replacement of filters, tubing etc).
- Switch on the electrostatic classifier (or other source of monodisperse particles), CPCs and VPR.
- Fill the CPCs with working fluid and allow the saturator and condenser to reach their specified temperatures.
- Apply an external vacuum source to the CPCs if not fitted with an internal pump.
- Do not proceed unless all indicators on the CPCs show correct instrument status (eg temperature, liquid level, flow and laser etc).
- Check the inlet flow rates of the CPCs with an appropriate calibrated flow meter (low pressure drop variety recommended, eg bubble flow meter).
- Check that zero concentration is reported when a HEPA filter is attached to the inlet of the CPCs. The CPC requires attention from the manufacturer if concentrations greater than 1 particles cm$^{-3}$ are reported.
- Apply clean dry filtered air to the VPR at pressures specified by the manufacturer.
- Switch on heating to VPR and allow to reach specified temperatures.
- Connect the CPCs to the VPR.
- When the VPR has reached its specified temperatures apply a HEPA filter to the inlet and ensure a zero concentration is reported on the CPC after the ET (eg PNC_GOLD). Do not proceed if particle concentrations of greater than 5 particles cm$^{-3}$ are reported.
Connect the inlet of PND₁ and the upstream CPC to the electrostatic classifier via a ‘Y’ connector, as illustrated in Figure 2.

Note: if an aerosol generator which produces monodisperse particles is used, the electrostatic classifier is not required and the VPR can be connected directly to the aerosol source.

- Ensure that the particle residence time in the pipework from the electrostatic classifier to both the upstream CPC and the inlet to PND₁ are identical. Take into account the inlet flow to each instrument and internal diameter and length of the interconnecting pipework. Take particular care if the inlet flow rates into the two instruments are different, this will require either different path lengths with pipework of the same internal diameter or the same path length with pipework of different diameters.
- Set the electrostatic classifier flows such that the monodisperse aerosol flow leaving the classifier is sufficient for the total inlet flow of both the CPC and the VPR (provide additional particle-free flow after the classifier if necessary).

Make arrangements to log data from both the upstream (inlet) and downstream (outlet) CPCs simultaneously at the same sampling rate.

Generate the calibration aerosol and connect the electrostatic classifier to the source.

- Select a monodisperse diameter using the electrostatic classifier and allow the number concentration to stabilise upstream of the VPR. All monodisperse particles must enter the VPR at concentrations of greater than 1000 particles cm\(^{-3}\). Once stable record measurements for 10 minutes up and downstream of the VPR (remember that not all CPCs are suitable for measuring 30nm diameter particles).
- Once a stable measurement has been taken the next monodisperse diameter should be selected on the electrostatic classifier and allowed to stabilise. This should be repeated until all monodisperse diameters have been measured.
- If you are using a CPC which is not suitable for measuring 30nm particles (eg PNC_GOLD) then use at least one that is capable of this measurement range and exchange its upstream and downstream sampling lines so that one CPC measures both the upstream and the downstream number concentrations. Each measurement must be stable and the upstream measurement must be verified after measuring the downstream concentration to ensure the inlet concentration has not drifted during the measurement, eg measure upstream concentration for 10 minutes, measure the downstream concentration for 10 minutes, then return to the upstream sampling arrangement and sample for 10 minutes to ensure the concentration is stable.

Once all required diameters have been measured the aerosol source should be disconnected from the VPR and a HEPA filter applied to the inlet to remove all particles from the system and to verify the
zero concentration leaving the VPR (should be less than 5 particle/cm³). This ensures the VPR has
not become contaminated from the calibration procedure.

4.2 Calibration of the VPR for volatile particle removal
efficiency (supression of nucleation particles)

The procedure for calibrating the VPR in terms of volatile particle removal efficiency is similar to that
described for solid particle penetration efficiency. There are a two key differences described below,
otherwise the procedure is identical:

- Only 1 monodisperse diameter is required, 30nm
- The inlet concentration to the VPR must be >10,000 particles cm⁻³.

4.3 Calculation of penetration efficiency of solid
particles

The penetration efficiencies (PE) should be calculated using the following equation:

\[
\frac{\text{Downstream number concentration}}{(\text{Upstream number concentration at PND}_1 \text{ inlet/ PND}_1 \text{ DF/ PND}_2 \text{ DF})} \times 100\% = \text{PE}
\]

*Note: Downstream number concentration is the number concentration at the outlet of the VPR ie the
PNC_GOLD sampling location,*

*Upstream number concentration is the number concentration at the inlet of the VPR ie the PND₁ inlet,*

*DF is dilution factor,*

*PND₁ DF is the dilution factor of the primary diluter and*

*PND₂ DF is the dilution factor of the secondary diluter.*

The dilution factors used for this calculation should be those supplied by the manufacturer’s most
recent gas calibration.

The number concentrations should be averaged over the 10 minute stable period.

100% penetration efficiency indicates zero particle losses in the system. The pass criteria is 80%
penetration efficiency at all diameters.
4.4 Calculation of suppression of nucleation particles

The average number concentration of volatile particles at the inlet of the VPR should be calculated over the 10 minute stable period. The performance requirement of the VPR is that >99% of tetracosane particles are removed. Therefore the pass criteria for the suppression of nucleation particles is 1% of the inlet concentration.

The average number concentration at the outlet of the VPR should be calculated over the 10 minute stable period, and if this is less than 1% of the inlet concentration then the VPR performance is acceptable.
5 References


2 Andersson JD, Clark D UN-GRPE PMP Phase 3 Inter-laboratory Correlation Exercise: Framework and Laboratory Guide, A Document For The UK Department for Transport. RD 04/80801.5

3 Marshall, IA Condensation Particle Counter Calibration Procedures, report to Department for Transport, netcen/ED44970001/Issue 2, September 2006.