

# Volatile Particle Remover Calibration and Validation Procedures

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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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and ISO14001

<b>Author</b>	Name	Emma Sandbach
<b>Approved by</b>	Name	Ian Marshall
	Signature	
	Date	19 December 2007



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# 1 Introduction

Appendix 5 of the 'Proposal for draft supplement 7 to the 05 series of amendments to Regulation No.83'<sup>1</sup> describes a sampling pre-conditioning system to be used for particle number emission measurements of emissions after a cold start for type approval testing. 

The sampling pre-conditioning unit as described is capable of diluting the sample in one or more stages to achieve a particle number concentration below the threshold of the single particle count mode of the particle number counter and to achieve a gas temperature below 35°C at the particle number counter. 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 version of the VPR described in the recommended system 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 and validation procedures for the VPR. Calibration of the VPR is to be performed at the point of manufacture and validation is to be performed at least at 6 month intervals for all instruments without automatic temperature sensors or at least at 12 month intervals if automatic temperature sensors are present. The calibration procedure must be repeated if the system 'fails' its validation procedure or the particle number measurement system is modified in any way. 

## 2 VPR Overview

### 2.1 Requirements

The specific requirements for the sample preconditioning unit are described in Appendix 5, Section 1.3 of 'Proposal for draft supplement 7 to the 05 series of amendments to Regulation No.83'<sup>1</sup>.

Note: bold text highlights the most recent changes (Oct 07) to this Appendix 

1.3.1. **The particle sample shall not pass through a pump before passing through the PNC.**

1.3.2. A sample pre-classifier is recommended.

1.3.3. The sample preconditioning unit shall:

1.3.3.1. Be capable of diluting the sample in one or more stages to achieve a particle number concentration below **the upper threshold of the single particle count mode of the PNC and a gas temperature below 35 °C at the inlet to the PNC.**

1.3.3.2. Include an initial heated dilution stage which outputs **a sample** at a temperature of **≥150°C and ≤400°C and dilutes by a factor of at least 10.**

1.3.3.3. **Achieve a particle concentration reduction factor (  $f_r(d_i)$  ), as defined in section 2.2.2, for particles of 30 nm<sup>a</sup> and 50 nm electrical mobility diameters, that is no more than 30 % and 20 % respectively higher, and no more than 5 % lower than that for particles of 100 nm electrical mobility diameter for the VPR as a whole.**

1.3.3.4. **Also achieve >99.0 % vaporisation of 30 nm tetracontane ( $\text{CH}_3(\text{CH}_2)_{38}\text{CH}_3$ ) particles, with an inlet concentration of  $\geq 10,000 \text{ cm}^{-3}$ , by means of heating and reduction of partial pressures of the tetracontane.**

### 2.2 Recommended System

The following section describes the recommended practice for particle number measurement, taken from Section 1.4, Appendix 5 of the 'Proposal for draft supplement 7 to the 05 series of amendments to Regulation No.83'<sup>1</sup>. However, any sample preconditioning system meeting the requirements detailed in Section 2.1 may be used. The recommended system is illustrated in Figure 1.

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<sup>a</sup> All particle diameters referred to in this document are electrical mobility diameters.

#### 1.4.4. Volatile Particle Remover

The VPR shall comprise one particle number diluter ( $PND_1$ ), an evaporation tube and a second diluter ( $PND_2$ ) in series. This dilution function is to reduce the number concentration of the sample entering the particle concentration measurement unit to less than **the upper threshold of the single particle count mode of the PNC** and to suppress nucleation within the sample.

The VPR shall achieve **>99.0 % vaporisation of 30 nm tetracontane ( $CH_3(CH_2)_{38}CH_3$ ) particles, with an inlet concentration of  $\geq 10,000 \text{ cm}^{-3}$ , by means of heating and reduction of partial pressures of the tetracontane. It shall also achieve a particle concentration reduction factor ( $f_r$ ) for particles of 30 nm and 50 nm electrical mobility diameters, that is no more than 30 % and 20 % respectively higher, and no more than 5 % lower than that for particles of 100 nm electrical mobility diameter for the VPR as a whole.**

##### 1.4.4.1. First Particle Number Dilution Device ( $PND_1$ )

The first particle number dilution device shall be specifically designed to dilute particle number concentration and operate at a (wall) temperature of  $150^\circ\text{C} - 400^\circ\text{C}$ . The wall temperature setpoint should not exceed the wall temperature of the ET (paragraph 1.4.4.2.). The diluter should be supplied with HEPA filtered dilution air and be capable of a dilution factor of 10 to **200** times.

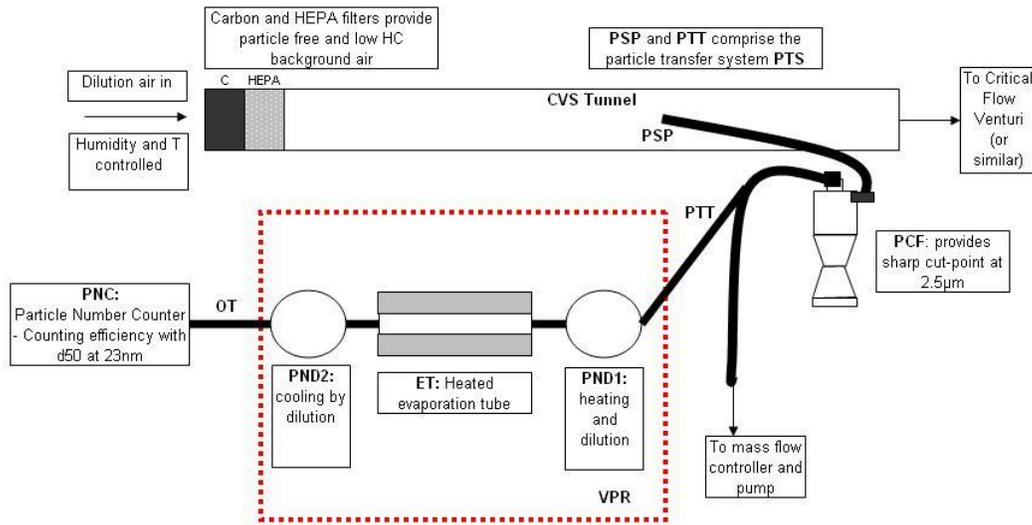
##### 1.4.4.2. Evaporation Tube

The entire length of the ET shall be controlled to a wall temperature greater than or equal to that of the first particle number dilution device and the wall temperature held at a fixed value between  $300^\circ\text{C}$  and **400**  $^\circ\text{C}$ .

##### 1.4.4.3. Second Particle Number Dilution Device ( $PND_2$ )

**$PND_2$**  shall be specifically designed to dilute particle number concentration. The diluter shall be supplied with HEPA filtered dilution air and be capable of maintaining a single dilution factor within a range of 10 to 30 times. The dilution factor of  $PND_2$  shall be selected in the range between 10 and 15 such that particle number concentration downstream of the second diluter **is less than the upper threshold of the single particle count mode of the PNC** and the gas temperature prior to entry to the PNC is  $<35^\circ\text{C}$ .

**Figure 1: Schematic of Recommended Particle Sampling System**



## 3 Primary Calibration Methods

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

- 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.
- The calibration methods may 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 whilst at the same time ensuring pathways are as short as possible to minimise losses by agglomeration. A simple method to ensure sample pathways have the same losses is to measure each sample line using one PNC at a high aerosol concentration and check the measured concentration is the same.
- Use of conductive tubing shall be required to minimise particle deposition.
- All calibrations shall be performed at room temperatures of  $\leq 35$  
- The term 'upstream' refers to the sampling location at the inlet of the VPR *i.e.* PND<sub>1</sub> inlet.
- The term 'downstream' refers to the sampling location at the outlet of the VPR *i.e.* after the second diluter PN 

### 3.1 Primary Calibration Criteria

Calibration of the VPR's particle concentration reduction factors across its full range of dilution settings, at the instrument manufacturer's recommended operating temperatures, shall be required when the unit is new and following any major maintenance. The periodic validation requirement for the VPRs particle concentration reduction factor is limited to a check at a single setting  typical of that used for measurement on diesel particulate filter equipped vehicles. The Technical Service shall ensure the existence of a calibration or validation certificate for the volatile particle remover within a 6 month period prior to the emissions test. If the volatile particle remover incorporates temperature monitoring alarms a 12 month validation interval shall be permissible.

Additionally, the Technical Service shall ensure the existence of a validation certificate for the VPR demonstrating effective volatile particle removal efficiency within a 6 month period prior to the

emissions test. If the volatile particle remover incorporates temperature monitoring alarms a 12 month validation interval shall be permissible.

### 3.1.1 Particle Concentration Reduction Factor ( $f_r$ )

Calibration shall be undertaken by measuring the particle concentration reduction factor ( $f_r$ ) of solid particles with an electrical mobility diameter of 30, 50 and 100 nm. For VPRs with a fixed dilution factor the particle concentration reduction factor should be measured at the fixed setting. For VPRs with a variable primary diluter ( $PND_1$ ) calibration shall be undertaken for at least 5 dilution factors spaced logarithmically across the nominal dilution factor range. In the case of VPRs with a variable secondary diluter ( $PND_2$ ) the calibration must be performed across a matrix of 15 nominal dilution settings (the 5 settings at  $PND_1$  and 3 settings (10, 15 and an intermediate setting) at  $PND_2$ ). At each nominal dilution setting it shall achieve a particle concentration reduction factor ( $f_r$ ) for particles of 30 nm and 50 nm electrical mobility diameters, that is no more than 30 % and 20 % respectively higher, and no more than 5 % lower than that for particles of 100 nm electrical mobility diameter for the VPR as a whole.

Calibration shall be conducted at the instrument manufacturer's recommended operating temperatures.

### 3.1.2 Volatile Particle Removal Efficiency

The VPR shall achieve >99.0 % vaporisation of 30 nm tetracontane ( $CH_3(CH_2)_{38}CH_3$ ) particles, with an inlet concentration of  $\geq 10,000 \text{ cm}^{-3}$ , by means of heating and reduction of partial pressures of the tetracontane when operated at the following conditions:

- Manufacturer's recommended evaporation tube temperature.
- VPRs with variable dilution factor settings: nominal secondary dilution factor of 10 ( $PND_2$ ), and the lowest  $PND_1$  nominal dilution setting.

## 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 100 nm diameter including condensation generators, combustion aerosol generators, nebulisation and hot wires. Some examples of these techniques are detailed in References 3,4,6 and 7. This is not an exhaustive list and any of these methods are suitable for producing calibration aerosols, but for the VPR calibration they must meet the following criteria:

- Capable of producing a stable aerosol at a minimum concentration of  $5,000 \text{ particles cm}^{-3}$  and below the single particle count mode limit of the particle number counter.
- Monodispersity:  $GSD \leq 1.2$ .

- Relative humidity <50 %.
- The aerosol generation technique and aerosol material must produce a solid aerosol that is free from volatile particles.

Any calibration aerosol must be stable at VPR operating conditions, i.e. it should be physically and chemically stable and its aerodynamic behaviour should not change at ET operating temperatures.



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



### 3.2.2 Volatile particles

The requirements for volatile particles are more prescriptive than for solid particles, these are:

- Tetracontane ( $\text{CH}_3(\text{CH}_2)_{38}\text{CH}_3$ ) particles with an electrical mobility diameter of at least 30 nm at a concentration  $\geq 10,000$  particles  $\text{cm}^{-3}$ .
- Monodispersity:  $\text{GSD} \leq 1.2$ .

A condensation generator is a simple method to produce these particles. The monodispersity of the aerosol (for both solid and volatile particles) should be verified if it is not defined by the aerosol generator itself. A technique for producing solid and volatile particles is described in the example of Annex 1. Alternative methods that generate similarly sized particles and concentrations are equally appropriate.

## 3.3 Additional equipment requirements

The following table details the equipment, along with their purposes, that are additional to the components of the particle number measurement system and are recommended for successful calibration of the volatile particle remover (Table 1).

**Table 1: Description of equipment required for calibration of the VPR**

<b>Required equipment</b>	<b>Purpose</b>
1 calibrated flow meter	Measurement of particle counter flow
1 HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency)	Zero checks on PNC and VPR
Aerosol generator(s)	To generate solid and volatile particles for VPR performance checks
1 neutraliser	Neutralisation of charges on aerosol for calibration
Calibrated Electrostatic Classifier/Scanning Mobility Particle Sizer	Production of monodisperse aerosols. Other instruments may be used to produce monodisperse particles but an SMPS may be required to verify the GSD.
<b><i>Optional</i></b>	
Additional calibrated PNC	For simultaneous measurements of upstream and downstream particle number concentration for VPR performance checks.
Calibrated flow meter and pump	To simulate downstream PNC flow from VPR whilst taking upstream measurements if calibrating the VPR with one PNC.

## 4 Validation Methods

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

- 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 validation is appropriately calibrated before use.
- The validation methods may 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 whilst at the same time ensuring pathways are as short as possible to minimise losses by agglomeration. A simple method to ensure sample pathways have the same losses is to measure each sample line using one PNC at a high aerosol concentration and check the measured concentration is the same.
- Use of conductive tubing shall be required to minimise particle deposition.
- All calibrations shall be performed at room temperatures of  $\leq 35^{\circ}\text{C}$ .
- The term 'upstream' refers to the sampling location at the inlet of the VPR *i.e.* PND<sub>1</sub> inlet.
- The term 'downstream' refers to the sampling location at the outlet of the VPR *i.e.* after the second diluter PND<sub>2</sub>.

### 4.1 Validation Method Criteria

The VPR must be validated in terms of its particle concentration reduction factor ( $f_r$ ) and its volatile particle removal efficiency (or suppression of nucleation particles).

The Technical Service shall ensure the existence of a validation certificate of the volatile particle remover within a 6-month period prior to the emissions test. If the volatile particle remover incorporates temperature monitoring alarms a 12-month validation interval shall be permissible.

The validation aerosol and equipment requirements are identical to those described for primary calibration in Sections 3.2 and 3.3.

An additional, optional quality control check of VPR performance may also be performed more frequently, as required, through measurement of the dilution factors using gas analyser. Table 2 describes the additional required equipment if this optional check is adopted.

**Table 2: Required equipment for optional quality control check with gas dilution**

Required equipment	Purpose
1 calibrated gas analyser (recommended CO, CO <sub>2</sub> or NO <sub>x</sub> analyser with converter)	Calibration of the diluters
1 bottle of certified gas (recommended ≥ 50,000 ppm CO, CO <sub>2</sub> , N <sub>2</sub> , propane or NO, uncertainty on certified concentration better than 2%)	Calibration of the diluters
1 calibrated flow meter/rotameter	To indicate inlet flow of gas supply to diluter to ensure ambient pressure at diluter inlet.

#### 4.1.1 Particle Concentration Reduction Factor ( $f_r$ )

Validation shall be undertaken by measuring the particle concentration reduction factor ( $f_r$ ) of solid particles with an electrical mobility diameter of 30, 50 and 100 nm. For VPRs with variable dilution factor settings validation shall be undertaken at one dilution setting typical for DPF exhaust emissions measurement and at the manufacturer's recommended operating temperatures.

The validation acceptance criterion is that the validation measurement is  $\pm 10\%$  of the particle concentration reduction factor ( $f_r$ ) as determined during the primary calibration.

#### 4.1.2 Volatile Particle Removal Efficiency

The VPR shall achieve >99.0 % vaporisation of 30 nm tetracontane ( $\text{CH}_3(\text{CH}_2)_{38}\text{CH}_3$ ) particles, with an inlet concentration of  $\geq 10,000 \text{ cm}^{-3}$ , by means of heating and reduction of partial pressures of the tetracontane when operated at the following conditions:

- Manufacturer's recommended evaporation tube temperature.
- VPRs with variable dilution factor settings: nominal secondary dilution factor of 10 (PND<sub>2</sub>), and the lowest PND<sub>1</sub> nominal dilution setting.

## 5 Primary Calibration Procedures

The procedures for performing a VPR calibration are described below. For all calibration and validation procedures a stable aerosol is defined as having an upstream concentration drift of less than 10 % over a 5 minute period 

### 5.1 Particle concentration reduction factor ( $f_r$ )

The particle concentration reduction factor ( $f_r$ ) may be measured in two ways:

1. Using one particle counter. In this case the upstream and downstream particle number concentrations are recorded using the same PNC sampling at either position through equivalent sampling lines. Upstream and downstream sampling lines are simply exchanged and the number concentration allowed to stabilise before measurement commences. Finally, the upstream sampling line should be reconnected to the PNC to verify that the upstream concentration has not drifted (by more than 10 %) during the measurement 
2. Using two particle counters, one measuring upstream/at the VPR inlet and one measuring downstream/at the VPR outlet. If this option is selected then it is assumed that the correlation between the two PNCs (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<sup>5</sup>' document. The PNCs' response at all particle diameters (30, 50 and 100 nm) must be measured. Readings from one PNC must be adjusted to take into consideration any differences between the two instruments. If two different models of PNCs are used, their counting efficiency may differ significantly for 30 nm diameter particles  Additionally the data from the upstream and downstream PNCs must be time aligned to account for the VPR residence time.



Prepare the VPR and PNCs for use:

- a. Position all apparatus according to the manufacturers instructions, this will be generally similar to that described in the example, Annex 1, Figure 2.
- b. Where appropriate clean any dilution mechanisms within the VPR as advised by the manufacturer (e.g. for rotating disc diluters remove the disc and clean using an appropriate solvent).
- c. Perform any routine maintenance of the VPR as advised by the manufacturer (e.g. replacement of filters, tubing *etc.*).
- d. Switch on the electrostatic classifier (or other source of monodisperse particles), PNCs and VPR.
- e. Fill the PNCs with working fluid and allow the saturator and condenser to reach their specified temperatures.
- f. Apply an external vacuum source to the PNCs if not fitted with an internal pump.
- g. Do not proceed unless all indicators on the PNCs show correct instrument status (e.g. temperature, liquid level, flow and laser *etc.*).

- h. Check the inlet flow rates of the PNCs with an appropriate calibrated flow meter (low pressure drop variety recommended, e.g. bubble flow meter). The flow into the particle counter shall report a measured value within 5 percent of the particle counter nominal flow rate.
- i. Check that zero concentration is reported when a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) is attached to the inlet of the PNCs. The PNC requires attention from the manufacturer if concentrations greater than 0.2 particles  $\text{cm}^{-3}$  are reported.
- j. If required, apply clean dry filtered air to the VPR at pressures specified by the manufacturer.
- k. Switch on heating to VPR and allow to reach specified temperatures.
- l. Connect the PNC to the VPR.
- m. When the VPR has reached its specified temperature settings connect a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) to the inlet and ensure a zero concentration is reported on the PNC. Do not proceed if particle concentrations greater than 0.5 particles  $\text{cm}^{-3}$  are reported.
- n. If the “one PNC” method is being used for the calibration exchange the sampling line from the VPR outlet location (downstream) to the VPR inlet (upstream) and use a mass flow controller, or similar, to simulate the flow rate of the PNC from the VPR. If the “two PNC” method is used split the flow from the neutraliser between the upstream PNC and the inlet of the VPR.

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

- o. Ensure that the particle residence time in the pipework from the neutraliser to both the upstream PNC and the inlet to the VPR are identical. Residence time must be adjusted by varying the length rather than the diameter of the pathway, as diffusional deposition is independent of tube diameter for a given volumetric flow rate.
- p. Set the electrostatic classifier flows such that the monodisperse aerosol flow leaving the classifier is sufficient for the total inlet flow of both the PNC and the VPR (provide additional particle-free flow after the classifier if necessary, ensuring an adequate mixing length for the aerosol and particle-free air before the inlet of the PNC and VPR).

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

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

- q. For variable primary diluters select a nominal dilution setting on PND<sub>1</sub>.  
For VPRs with fixed dilution factors skip to step (s).
- r. For variable PND<sub>2</sub> diluters select a nominal dilution setting on PND<sub>2</sub>.
- s. Select a monodisperse diameter (30, 50 or 100 nm) 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 5,000 particles  $\text{cm}^{-3}$ . Once stable record

- measurements for 1 minute (or the time required for a cumulative number count of 10,000 particles, whichever is greater) up and downstream of the VPR
- t. For VPRs with fixed dilution factors skip to step (x).  
For VPRs with variable dilution factors: Once a stable measurement has been taken select the next nominal dilution setting on PND<sub>1</sub>, allow to stabilise and record measurements for 1 minute (or the time required for a cumulative number count of 10,000 particles, whichever is greater) up and downstream of the VPR.
  - u. Repeat step (t) until all five dilution settings of PND<sub>1</sub> have been measured.
  - v. For variable PND<sub>2</sub> diluters, select the next nominal dilution setting, and repeat the measurements for all five dilution settings on PND<sub>1</sub>.
  - w. Select the third and final nominal dilution setting on PND<sub>2</sub> and repeat the measurements for all five dilution settings on PND<sub>1</sub>.

*Note: The minimum number of measurement conditions per particle diameter should be 15 for a variable secondary diluter, for a fixed secondary diluter the minimum number of measurement conditions should be five per particle diameter. For VPRs with fixed primary and secondary dilution factors only 1 measurement is required at each particle diameter.*

- x. The next monodisperse diameter should be selected on the electrostatic classifier and allowed to stabilise. Repeat steps (s) to (w) for each monodisperse diameter.
- y. If the “one PNC” method is used 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, e.g. measure upstream concentration for 1 minute, measure the downstream concentration for 1 minute, then return to the upstream sampling arrangement and sample for 1 minute to ensure the concentration is stable (or the time required for a cumulative number count of 10,000 particles, whichever is greater). Measurement is valid if upstream particle concentration is within  $\pm 10\%$  of initial reading.

*Note: Sample times must be sufficient to ensure a minimum of 10,000 particles are counted at each measurement condition (cumulatively over the sample period).*

Once all required diameters and nominal dilution settings have been measured the aerosol source should be disconnected from the VPR and a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) applied to the inlet to remove all particles from the system and to verify the zero concentration leaving the VPR (should be less than  $0.5 \text{ particle cm}^{-3}$ ). This ensures the VPR has not become contaminated from the calibration procedure.

## 5.2 Validation of the VPR for volatile particle removal efficiency

For this validation only one monodisperse diameter is required; tetracontane particles ( $\text{CH}_3(\text{CH}_2)_{38}\text{CH}_3$ ) of electrical mobility diameter of at least 30 nm at an inlet concentration of  $\geq 10,000$  particles  $\text{cm}^{-3}$ .

VPRs with variable dilution settings should be validated at one nominal dilution setting, with  $\text{PND}_1$  at the lowest setting and for variable  $\text{PND}_2$  diluters a nominal dilution setting of 10 should be used. The evaporation tube should be at the operating conditions recommended by the manufacturer (and that most commonly used for DPF exhaust emissions measurements).

There are two methods for measurement of volatile particle removal efficiency:

### Method 1 - Measurement of up and downstream particle concentrations using a PNC

This can be the PNC supplied with the particle number measurement system. For the volatile particle removal efficiency calibration an upstream concentration of  $\geq 10,000$  particles  $\text{cm}^{-3}$  is required, however this concentration may be above the full-scale deflection of the PNC in single particle count mode. An assumption can be made that any measurement by the PNC above this limit ensures the VPR is challenged with a minimum of 10,000 particles  $\text{cm}^{-3}$ . The procedure for this method is as follows (this validation can be performed with either one or two PNCs, if two PNCs are used their response at all particle diameters (30, 50 and 100 nm) must be compared using the secondary calibration method described in the 'Condensation Particle Counter Calibration Procedures'<sup>5</sup> document. One PNC must be adjusted to take into consideration any differences between the two instruments. If two different models of PNC are used their counting efficiency may differ significantly for 30 nm diameter particles. Additionally the data from the upstream and downstream PNCs must be time aligned to account for the VPR residence time):

Prepare the VPR and PNCs for use:

- Position all apparatus according to the manufacturers instructions, this will be generally similar to that described in the example, Annex 1, Figure 2.
- Where appropriate clean any dilution mechanisms within the VPR as advised by the manufacturer (e.g. 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 (e.g. replacement of filters, tubing etc.).
- Switch on the electrostatic classifier (or other source of monodisperse particles), PNCs and VPR.
- Fill the PNCs with working fluid and allow the saturator and condenser to reach their specified temperatures.
- Apply an external vacuum source to the PNCs if not fitted with an internal pump.

- g. Do not proceed unless all indicators on the PNCs show correct instrument status (e.g. temperature, liquid level, flow and laser etc.).
- h. Check the inlet flow rates of the PNCs with an appropriate calibrated flow meter (low pressure drop variety recommended, e.g. bubble flow meter). The flow into the particle counter shall report a measured value within 5 percent of the particle counter nominal flow rate.
- i. Check that zero concentration is reported when a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) is attached to the inlet of the PNCs. The PNC requires attention from the manufacturer if concentrations greater than  $0.2 \text{ particles cm}^{-3}$  are reported.
- j. If required, apply clean dry filtered air to the VPR at pressures specified by the manufacturer.
- k. Switch on heating to VPR and allow to reach specified temperatures.
- l. Connect the PNC to the VPR.
- m. When the VPR has reached its specified temperature settings connect a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) to the inlet and ensure a zero concentration is reported on the PNC. Do not proceed if particle concentrations greater than  $0.5 \text{ particles cm}^{-3}$  are reported.
- n. If the “one PNC” method is used for the volatile particle calibration exchange the sampling line from the VPR outlet location (downstream) to the VPR inlet (upstream) and use a mass flow controller, or similar, to simulate the flow rate of the PNC from the VPR. If the “two PNC” method is adopted split the flow from the neutraliser between the upstream PNC and the inlet of the VPR.

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

- o. Ensure that the particle residence time in the pipework from the neutraliser to both the upstream PNC and the inlet to the VPR are identical. Residence time must be adjusted by varying the length rather than the diameter of the pathway, as diffusional deposition is independent of tube diameter for a given volumetric flow rate.
- p. Set the electrostatic classifier flows such that the monodisperse aerosol flow leaving the classifier is sufficient for the total inlet flow of both the PNC and the VPR (provide additional particle-free flow after the classifier if necessary, ensuring an adequate mixing length for the aerosol and particle-free air before the inlet of the PNC and VPR).

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

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

- q. Select 30 nm diameter particles using the electrostatic classifier and allow the number concentration to stabilise upstream of the VPR. Once stable record measurements for 1 minute (or the time required for a cumulative number count of 10,000 particles, whichever is greater) up and downstream of the VPR.

- r. If the “one PNC” method is adopted 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, e.g. measure upstream concentration for 1 minute, measure the downstream concentration for 1 minute, then return to the upstream sampling arrangement and sample for 1 minute to ensure the concentration is stable (or the time required for a cumulative number count of 10,000 particles, whichever is greater). The drift of the upstream aerosol concentration must not be more than 10 %.

Once complete the aerosol source should be disconnected from the VPR and a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) applied to the inlet to remove all particles from the system and to verify the zero concentration leaving the VPR (should be less than 0.5 particle cm<sup>-3</sup>). This ensures the VPR has not become contaminated from the validation procedure.

### Method 2 - One PNC measuring downstream of the VPR only

The particle counter used for this method can be the PNC supplied with the particle number measurement system. The procedure for this method is as follows:

Prepare the VPR and PNCs for use:

- a. Position all apparatus according to the manufacturers instructions, this should be something similar to that described in the example, Annex 1, Figure 2.
- b. Where appropriate clean any dilution mechanisms within the VPR as advised by the manufacturer (e.g. for rotating disc diluters remove the disc and clean using an appropriate solvent).
- c. Perform any routine maintenance of the VPR as advised by the manufacturer (e.g. replacement of filters, tubing etc.).
- d. Switch on the electrostatic classifier (or other source of monodisperse particles), PNC and VPR.
- e. Fill the PNC with working fluid and allow the saturator and condenser to reach their specified temperatures.
- f. Apply an external vacuum source to the PNC if not fitted with an internal pump.
- g. Do not proceed unless all indicators on the PNC show correct instrument status (e.g. temperature, liquid level, flow and laser etc.).
- h. Check the inlet flow rates of the PNC with an appropriate calibrated flow meter (low pressure drop variety recommended, e.g. bubble flow meter). The flow into the particle counter shall report a measured value within 5 percent of the particle counter nominal flow rate.
- i. Check that zero concentration is reported when a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) is attached to the inlet of the PNC. The PNC requires attention from the manufacturer if concentrations greater than 0.2 particles cm<sup>-3</sup> are reported.
- j. If required, apply clean dry filtered air to the VPR at pressures specified by the manufacturer.
- k. Connect the PNC to the VPR.
- l. Do not heat the VPR, leave it at room temperature (upper limit of 35°C).

- m. Connect a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) to the inlet of the VPR and ensure a zero concentration is reported on the PNC. Do not proceed if particle concentrations of greater than  $0.5 \text{ particles cm}^{-3}$  are reported.

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

- n. Set the electrostatic classifier flows such that the monodisperse aerosol flow leaving the classifier is sufficient for the inlet flow of the VPR (provide additional particle-free flow after the classifier if necessary, ensuring an adequate mixing length for the aerosol and particle-free air before the inlet of the PNC and VPR).

Make arrangements to log data from the PNC.

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

- o. Select 30 nm using the electrostatic classifier and allow to stabilise for at least 1 minute (or the time required for a cumulative number count of 10,000 particles, whichever is greater). The monodisperse particles must enter the VPR at concentrations of greater than 10,000 particles  $\text{cm}^{-3}$ . Once stable record measurements for 1 minute downstream of the VPR (or the time required for a cumulative number count of 10,000 particles, whichever is greater).
- p. Switch on heating to VPR and allow to reach specified temperatures. Identical nominal dilution settings should be used at both temperature settings.
- q. Continue to supply 30 nm tetracontane particles to the VPR and record the particle concentration from the PNC (at the downstream location).
- r. When a stable downstream concentration is achieved, and the VPR has reached its temperature settings, record the downstream number concentrations over a 1 minute period (or the time required for a cumulative number count of 10,000 particles, whichever is greater).

Once complete the aerosol source should be disconnected from the VPR and a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) applied to the inlet to remove all particles from the system and to verify the zero concentration leaving the VPR (should be less than  $0.5 \text{ particle cm}^{-3}$ ). This ensures the VPR has not become contaminated from the validation procedure.

### **5.3 Calculation of particle concentration reduction factor ( $f_r$ )**

For each nominal dilution setting and particle diameter the ratio of upstream number concentration to downstream number concentration should be calculated:

The particle concentration reduction factor at each particle size ( $f_r(d_i)$ ) shall be calculated as follows;

$$f_r(d_i) = \frac{N_{in}(d_i)}{N_{out}(d_i)} \quad (1)$$

Where  $N_{in}(d_i)$  = upstream particle number concentration for particles of diameter  $d_i$ ;  
 $N_{out}(d_i)$  = downstream particle number concentration for particles of diameter  $d_i$ ; and  
 $d_i$  = particle electrical mobility diameter (30, 50 or 100 nm)

For each nominal dilution setting the mean particle concentration reduction factor ( $\overline{f_r}$ ) should then be calculated for all particle diameters;

$$\overline{f_r} = \frac{f_r(30 \text{ nm}) + f_r(50 \text{ nm}) + f_r(100 \text{ nm})}{3} \quad (2)$$

At each nominal dilution setting the VPR shall achieve a particle concentration reduction factor ( $f_r$ ) for particles of 30 nm and 50 nm electrical mobility diameters, that is no more than 30 % and 20 % respectively higher, and no more than 5 % lower than that for particles of 100 nm electrical mobility diameter for the VPR as a whole:

$$1.3 > \frac{f_r(30 \text{ nm})}{f_r(100 \text{ nm})} > 0.95 \quad (3)$$

$$1.2 > \frac{f_r(50 \text{ nm})}{f_r(100 \text{ nm})} > 0.95 \quad (4)$$

For VPRs with variable primary diluter settings a minimum of five particle concentration reduction factors ( $f_r$ ) corresponding to five nominal dilution settings are required. For VPRs with a variable secondary diluter (PND<sub>2</sub>) fifteen particle concentration reduction factors ( $f_r$ ) will be produced (five primary diluter settings at three secondary diluter settings). For VPRs with fixed dilution settings only one particle concentration reduction factor will be produced.

Some examples of particle concentration reduction factors are shown in Table 5, Annex 2.

## 5.4 Calculation of removal efficiency of volatile particles

There are two methods to calculate the removal efficiency of volatile particles depending on which validation method was adopted:

### 5.4.1 Method 1

The average number concentration of volatile particles at the inlet of the VPR should be calculated over the 1 minute stable period (or the time required for a cumulative number count of 10,000 particles, whichever is greater). The performance requirement of the VPR is that >99.0 % of tetracontane particles are removed. Therefore the pass criterion 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 1 minute stable period (or the time required for a cumulative number count of 10,000 particles, whichever is greater), and if this is less than 1 % of the inlet concentration then the VPR performance is acceptable.

A particle concentration reduction factor ( $f_r$ ) must be applied to the downstream number concentration measurements. This factor is that calculated at 30 nm, for the same nominal dilution settings used in the primary calibration with solid particles.

Volatile Particle Removal Efficiency Criteria:

$$\text{Average Downstream Concentration} \cdot f_r(30 \text{ nm}) \leq \frac{\text{Average Upstream Concentration}}{100} \quad (5)$$

where  $f_r(30 \text{ nm})$  = particle concentration reduction factor at 30 nm and the same nominal dilution settings.

#### 5.4.2 Method 2

The average number concentration of volatile particles downstream of the VPR should be calculated over the 1 minute stable period (or the time required for a cumulative number count of 10,000 particles, whichever is greater) for both temperature conditions i.e. room temperature and heated.

Volatile Particle Removal Efficiency Acceptance Criteria:

$$\text{Average Downstream Concentration (HT)} \leq \frac{\text{Average Downstream Concentration (RT)}}{100} \quad (6)$$

where RT = room temperature VPR  
HT = heated VPR

## 6 Validation Procedures

The procedures for performing a VPR validation are described below. For all procedures a stable aerosol is defined as having an upstream concentration drift of less than 10 % over a 5 minute period.

### 6.1 Particle concentration reduction factor ( $f_r$ )

The particle concentration reduction factor ( $f_r$ ) may be measured in two ways:

1. Using one particle counter. In this case the upstream and downstream particle number concentrations are recorded using the same PNC sampling at either position through equivalent sampling lines. Upstream and downstream sampling lines are simply exchanged and the number concentration allowed to stabilise before measurement commences. Finally, the upstream sampling line should be reconnected to the PNC to verify that the upstream concentration has not drifted by more than 10 % during the measurement.
2. Using two particle counters, one measuring upstream/at the VPR inlet and one measuring downstream/at the VPR outlet. If this option is selected then it is assumed that the correlation between the two PNCs (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<sup>5</sup>' document. The PNC's response at all particle diameters (30, 50 and 100 nm) must be measured. Readings from one PNC must be adjusted to take into consideration any differences between the two instruments. If using two different models of PNC, their counting efficiency may differ significantly for 30 nm diameter particles. Additionally the data from the upstream and downstream PNCs must be time aligned to account for the VPR residence time.

Prepare the VPR and PNCs for use:

- a. Position all apparatus according to the manufacturers instructions, this will be generally similar to that described in the example, Annex 1, Figure 2.
- b. Where appropriate clean any dilution mechanisms within the VPR as advised by the manufacturer (*e.g.* for rotating disc diluters remove the disc and clean using an appropriate solvent).
- c. Perform any routine maintenance of the VPR as advised by the manufacturer (*e.g.* replacement of filters, tubing *etc.*).
- d. Switch on the electrostatic classifier (or other source of monodisperse particles), PNCs and VPR.
- e. Fill the PNCs with working fluid and allow the saturator and condenser to reach their specified temperatures.
- f. Apply an external vacuum source to the PNCs if not fitted with an internal pump.

- g. Do not proceed unless all indicators on the PNCs show correct instrument status (e.g. temperature, liquid level, flow and laser etc.).
- h. Check the inlet flow rates of the PNCs with an appropriate calibrated flow meter (low pressure drop variety recommended, e.g. bubble flow meter). The flow into the particle counter shall report a measured value within 5 percent of the particle counter nominal flow rate.
- i. Check that zero concentration is reported when a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) is attached to the inlet of the PNCs. The PNC requires attention from the manufacturer if concentrations greater than  $0.2 \text{ particles cm}^{-3}$  are reported.
- j. If required, apply clean dry filtered air to the VPR at pressures specified by the manufacturer.
- k. Switch on heating to VPR and allow to reach specified temperatures.
- l. Connect the PNC to the VPR.
- m. When the VPR has reached its specified temperature settings connect a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) to the inlet and ensure a zero concentration is reported on the PNC. Do not proceed if particle concentrations greater than  $0.5 \text{ particles cm}^{-3}$  are reported.
- n. If the “one PNC” method is adopted for the validation exchange the sampling line from the VPR outlet location (downstream) to the VPR inlet (upstream) and use a mass flow controller, or similar, to simulate the flow rate of the PNC from the VPR. If the “two PNC” method is adopted split the flow from the neutraliser between the upstream PNC and the inlet of the VPR.

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

- o. Ensure that the particle residence time in the pipework from the neutraliser to both the upstream PNC and the inlet to the VPR are identical. Residence time must be adjusted by varying the length rather than the diameter of the pathway, as diffusional deposition is independent of tube diameter for a given volumetric flow rate.
- p. Set the electrostatic classifier flows such that the monodisperse aerosol flow leaving the classifier is sufficient for the total inlet flow of both the PNC and the VPR (provide additional particle-free flow after the classifier if necessary, ensuring an adequate mixing length for the aerosol and particle-free air before the inlet of the PNC and VPR).

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

- q. Generate the validation aerosol and connect the electrostatic classifier to the source.
- r. For VPRs with variable dilution settings select nominal dilution settings on PND<sub>1</sub> and PND<sub>2</sub> typically used for DPF exhaust emissions measurements.
- s. Select a monodisperse diameter (30, 50 or 100 nm) 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  $5000 \text{ particles cm}^{-3}$ . Once stable record

- measurements for 1 minute (or the time required for a cumulative number count of 10,000 particles, whichever is greater) up and downstream of the VPR
- t. The next monodisperse diameter should then be selected on the electrostatic classifier and allowed to stabilise. Repeat step (s) for each monodisperse diameter.
  - u. If the “one PNC” method is adopted 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, e.g. measure upstream concentration for 1 minute, measure the downstream concentration for 1 minute, then return to the upstream sampling arrangement and sample for 1 minute to ensure the concentration is stable (or the time required for a cumulative number count of 10,000 particles, whichever is greater). Measurement is valid if upstream particle concentration is within  $\pm 10\%$  of initial reading.

Once all required diameters have been measured the aerosol source should be disconnected from the VPR and a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) applied to the inlet to remove all particles from the system and to verify the zero concentration leaving the VPR (should be less than  $0.5 \text{ particle cm}^{-3}$ ). This ensures the VPR has not become contaminated from the calibration procedure.

## 6.2 Validation of the VPR for volatile particle removal efficiency

This validation requires tetracontane ( $\text{CH}_3(\text{CH}_2)_{38}\text{CH}_3$ ) particles of at least 30 nm electrical mobility diameter at an inlet concentration at the VPR of  $\geq 10,000 \text{ particles cm}^{-3}$ .

The VPR should be validated at one nominal dilution setting,  $\text{PND}_1$  should be at the lowest setting and for variable  $\text{PND}_2$  diluters a nominal dilution setting of 10 should be used. The evaporation tube should be at the operating conditions recommended by the manufacturer (and that most commonly used for DPF exhaust emissions measurements).

There are two methods for measurement of volatile particle removal efficiency:

### Method 1 - Measurement of up and downstream particle concentrations using a PNC

This can be the PNC supplied with the particle number measurement system. For the volatile particle removal efficiency validation an upstream concentration of  $\geq 10,000 \text{ particles cm}^{-3}$  is required, however this concentration will result in coincidence errors to be introduced into the PNC measurement. An assumption can be made that any measurement by the PNC above this limit ensures the VPR is challenged with a minimum of  $10,000 \text{ particles cm}^{-3}$ . This validation can be performed with either one or two PNCs. If two PNCs are used, their response at all particle diameters (30, 50 and 100 nm) must be measured using the secondary calibration method described in the ‘Condensation Particle Counter Calibration Procedures<sup>5</sup>’ document. One PNC must be adjusted to take into consideration any

differences between the two instruments. If two different models of PNC are used please note their counting efficiency may differ significantly for 30 nm diameter particles. Additionally the data from the upstream and downstream PNCs must be time aligned to account for the VPR residence time. The procedure for this method is as follows.

Prepare the VPR and PNCs for use:

- a. Position all apparatus according to the manufacturers instructions, this will be generally similar to that described in the example, Annex 1, Figure 2.
- b. Where appropriate clean any dilution mechanisms within the VPR as advised by the manufacturer (*e.g.* for rotating disc diluters remove the disc and clean using an appropriate solvent).
- c. Perform any routine maintenance of the VPR as advised by the manufacturer (*e.g.* replacement of filters, tubing *etc.*).
- d. Switch on the electrostatic classifier (or other source of monodisperse particles), PNCs and VPR.
- e. Fill the PNCs with working fluid and allow the saturator and condenser to reach their specified temperatures.
- f. Apply an external vacuum source to the PNCs if not fitted with an internal pump.
- g. Do not proceed unless all indicators on the PNCs show correct instrument status (*e.g.* temperature, liquid level, flow and laser *etc.*).
- h. Check the inlet flow rates of the PNCs with an appropriate calibrated flow meter (low pressure drop variety recommended, *e.g.* bubble flow meter). The flow into the particle counter shall report a measured value within 5 percent of the particle counter nominal flow rate.
- i. Check that zero concentration is reported when a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) is attached to the inlet of the PNCs. The PNC requires attention from the manufacturer if concentrations greater than 0.2 particles  $\text{cm}^{-3}$  are reported.
- j. If required, apply clean dry filtered air to the VPR at pressures specified by the manufacturer.
- k. Switch on heating to VPR and allow to reach specified temperatures.
- l. Connect the PNC to the VPR.
- m. When the VPR has reached its specified temperature settings connect a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) to the inlet and ensure a zero concentration is reported on the PNC. Do not proceed if particle concentrations greater than 0.5 particles  $\text{cm}^{-3}$  are reported.
- n. If the “one PNC” method is adopted for the volatile particle validation, exchange the sampling line from the VPR outlet location (downstream) to the VPR inlet (upstream) and use a mass flow controller, or similar, to simulate the flow rate of the PNC from the VPR. If the “two PNC” method is adopted split the flow from the neutraliser between the upstream PNC and the inlet of the VPR.

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

- o. Ensure that the particle residence time in the pipework from the neutraliser to both the upstream PNC and the inlet to the VPR are identical. Residence time must be adjusted by varying the length rather than the diameter of the pathway, as diffusional deposition is independent of tube diameter for a given volumetric flow rate.
- p. Set the electrostatic classifier flows such that the monodisperse aerosol flow leaving the classifier is sufficient for the total inlet flow of both the PNC and the VPR (provide additional particle-free flow after the classifier if necessary, ensuring an adequate mixing length for the aerosol and particle-free air before the inlet of the PNC and VPR).

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

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

- q. Select 30 nm diameter particles using the electrostatic classifier and allow the number concentration to stabilise upstream of the VPR. Once stable record measurements for 1 minute up and downstream of the VPR (or the time required for a cumulative number count of 10,000 particles, whichever is greater).
- r. If the “one PNC” method is adopted 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, *e.g.* measure upstream concentration for 1 minute, measure the downstream concentration for 1 minute, then return to the upstream sampling arrangement and sample for 1 minute to ensure the concentration is stable (or the time required for a cumulative number count of 10,000 particles, whichever is greater). The drift of the upstream aerosol concentration must be no more than 10 %.

Once complete the aerosol source should be disconnected from the VPR and a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) applied to the inlet to remove all particles from the system and to verify the zero concentration leaving the VPR (should be less than 0.5 particle cm<sup>-3</sup>). This ensures the VPR has not become contaminated from the calibration procedure.

### **Method 2 - One PNC measuring downstream of the VPR only**

The particle counter used for this method can be the PNC supplied with the particle number measurement system. The procedure for this method is as follows:

Prepare the VPR and PNCs for use:

- a. Position all apparatus according to the manufacturers instructions, this should be something similar to that described in the example, Annex 1, Figure 2.

- b. Where appropriate clean any dilution mechanisms within the VPR as advised by the manufacturer (e.g. for rotating disc diluters remove the disc and clean using an appropriate solvent).
- c. Perform any routine maintenance of the VPR as advised by the manufacturer (e.g. replacement of filters, tubing etc.).
- d. Switch on the electrostatic classifier (or other source of monodisperse particles), PNC and VPR.
- e. Fill the PNC with working fluid and allow the saturator and condenser to reach their specified temperatures.
- f. Apply an external vacuum source to the PNC if not fitted with an internal pump.
- g. Do not proceed unless all indicators on the PNC show correct instrument status (e.g. temperature, liquid level, flow and laser etc.).
- h. Check the inlet flow rates of the PNC with an appropriate calibrated flow meter (low pressure drop variety recommended, e.g. bubble flow meter). The flow into the particle counter shall report a measured value within 5 percent of the particle counter nominal flow rate.
- i. Check that zero concentration is reported when a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) is attached to the inlet of the PNC. The PNC requires attention from the manufacturer if concentrations greater than  $0.2 \text{ particles cm}^{-3}$  are reported.
- j. If required, apply clean dry filtered air to the VPR at pressures specified by the manufacturer.
- k. Connect the PNC to the VPR.
- l. Do not heat the VPR, leave it at room temperature (upper limit of  $35^{\circ}\text{C}$ ).
- m. Connect a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) to the inlet of the VPR and ensure a zero concentration is reported on the PNC. Do not proceed if particle concentrations of greater than  $0.5 \text{ particles cm}^{-3}$  are reported.

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

- n. Set the electrostatic classifier flows such that the monodisperse aerosol flow leaving the classifier is sufficient for the inlet flow of the VPR (provide additional particle-free flow after the classifier if necessary, ensuring an adequate mixing length for the aerosol and particle-free air before the inlet of the PNC and VPR).

Make arrangements to log data from the PNC.

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

- o. Select 30 nm using the electrostatic classifier and allow to stabilise for at least 1 minute (or the time required for a cumulative number count of 10,000 particles, whichever is greater). The monodisperse particles must enter the VPR at concentrations of greater than 10,000 particles  $\text{cm}^{-3}$ . Once stable record measurements for 1 minute downstream of the VPR (or the time required for a cumulative number count of 10,000 particles, whichever is greater).

- p. Switch on heating to VPR and allow to reach specified temperatures. Identical nominal dilution settings should be used at both temperature settings.
- q. Continue to supply 30 nm tetracontane particles to the VPR and record the particle concentration from the PNC (at the downstream location).
- r. When a stable downstream concentration is achieved, and the VPR has reached its temperature settings, record the downstream number concentration over a 1 minute period (or the time required for a cumulative number count of 10,000 particles, whichever is greater).

Once complete the aerosol source should be disconnected from the VPR and a HEPA filter (class H13 of EN 1822:1998 or better filtration efficiency) applied to the inlet to remove all particles from the system and to verify the zero concentration leaving the VPR (should be less than  $0.5 \text{ particle cm}^{-3}$ ). This ensures the VPR has not become contaminated from the calibration procedure.

### 6.3 Optional quality control check using gas dilution

A VPR quality control check should be performed under typical measurements conditions (*e.g.* those settings used to achieve particle concentration reduction factors,  $f_r = 150$ ,  $f_r = 1500$ ) and at the instrument manufacturer's recommended operation temperatures, in order to measure the corresponding gas concentration reduction factors.

The upstream measurement of the undiluted gas concentration is not required if a certified gas is being used for the diluter quality control check. The only measurement that needs to be made is the diluted gas concentration downstream of the VPR.

Gas Concentration Reduction Factor Measurement Procedure:

Prepare the particle number system (VPR) for normal use.

- a. Where appropriate clean any dilution mechanisms within the VPR as advised by the manufacturer
- b. Perform any routine maintenance of the VPR as advised by the manufacturer (*e.g.* replacement of filters, tubing *etc.*).
- c. Prepare the appropriate calibrated gas analyser for use, following manufacturers guidance for stabilisation/warm up period.
- d. Zero and span the analyser according to the manufacturers instructions.
- e. Connect the gas analyser to the VPR outlet (PNC position), ensuring that flow rates in the VPR meet the manufacturer's specifications.

Supply the VPR with gas ensuring that the system does not become over pressurised. This can easily be achieved with a flow splitter at the inlet of the VPR to provide gas at ambient pressure to the first diluter while using a flow meter to confirm that excess gas is vented to an exhaust or by using a bag evacuated and then filled with the certified gas.

- f. Select the first dilution setting for checking and begin recording data from the analyser. Allow the downstream measurement to stabilise and record data for at least 2 minutes. Once a stable measurement has been recorded select the next dilution setting and allow to stabilise. Repeat this step for at least one other dilution setting.
- g. If the VPR comprises more than one diluter, they must be measured as a complete system under the instrument manufacturer's recommended operation conditions.

Once the gas concentration reduction factor check is complete, then the analyser zero and span should be repeated to ensure analyser performance has not drifted during the procedure. The check is considered acceptable if the difference between the two zero and span measurements is less than 2 percent.

## 6.4 Calculation of particle concentration reduction factor ( $f_r$ )

The particle concentration reduction factor at each particle size ( $f_r(d_i)$ ) shall be calculated as follows;

$$f_r(d_i) = \frac{N_{in}(d_i)}{N_{out}(d_i)} \quad (7)$$

Where  $N_{in}(d_i)$  = upstream particle number concentration for particles of diameter  $d_i$ ;  
 $N_{out}(d_i)$  = downstream particle number concentration for particles of diameter  $d_i$ ; and  
 $d_i$  = particle electrical mobility diameter (30, 50 or 100 nm)

The mean particle concentration reduction factor ( $\overline{f_r}$ ) should then be calculated for all particle diameters:

$$\overline{f_r} = \frac{f_r(30nm) + f_r(50nm) + f_r(100nm)}{3} \quad (8)$$

The requirement for successful validation of the VPR is that the measured mean ( $\overline{f_r}$ ) is  $\pm 10\%$  of the mean particle concentration reduction factor ( $\overline{f_r}$ ) measured during the primary calibration (at the same nominal dilution settings).

If this requirement has not been met, the primary calibration should be repeated.

## 6.5 Calculation of removal efficiency of volatile particles

There are two methods to calculate the removal efficiency of volatile particle depending on which validation method was adopted:

### 6.5.1 Method 1

The average number concentration of volatile particles at the inlet of the VPR should be calculated over the 1 minute stable period (or the time required for a cumulative number count of 10,000 particles, whichever is greater). The performance requirement of the VPR is that >99.0 % of tetracontane particles are removed. Therefore the pass criterion 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 1 minute (or the time required for a cumulative number count of 10,000 particles, whichever is greater) stable period, and if this is less than 1 % of the inlet concentration then the VPR performance is acceptable.

A particle concentration reduction factor ( $f_r$ ) must be applied to the downstream number concentration measurements. This factor is that calculated at 30 nm, for the same nominal dilution settings used in the primary calibration with solid particles.

Volatile Particle Removal Efficiency Criteria:

$$\text{Average Downstream Concentration} \cdot f_r(30 \text{ nm}) \leq \frac{\text{Average Upstream Concentration}}{100} \quad (9)$$

where  $f_r(30 \text{ nm})$  = particle concentration reduction factor at 30 nm and the same nominal dilution settings.

### 6.5.2 Method 2

The average number concentration of volatile particles downstream of the VPR should be calculated over the 1 minute (or the time required for a cumulative number count of 10,000 particles, whichever is greater) stable period for both temperature conditions *i.e.* room temperature and heated.

Volatile Particle Removal Efficiency Acceptance Criteria:

$$\text{Average Downstream Concentration (HT)} \leq \frac{\text{Average Downstream Concentration (RT)}}{100} \quad (10)$$

where RT = room temperature VPR  
HT = heated VPR

## 6.6 Calculation of the quality control check gas concentration reduction factors

Gas concentration reduction factors from the quality control check should be calculated from the average downstream gas concentration measurements.

$$\text{Gas Concentration Reduction Factor} = \frac{\text{Certified Gas Concentration (ppm)}}{\text{Average Downstream Gas Concentration (ppm)}} (1) $$

The gas concentration reduction factor calculation should be performed for both dilution conditions measured.

The maximum permissible deviation between the gas concentration reduction factors measured at the time of the primary calibration and those measured during the quality control check should be within less than 5 per cent.

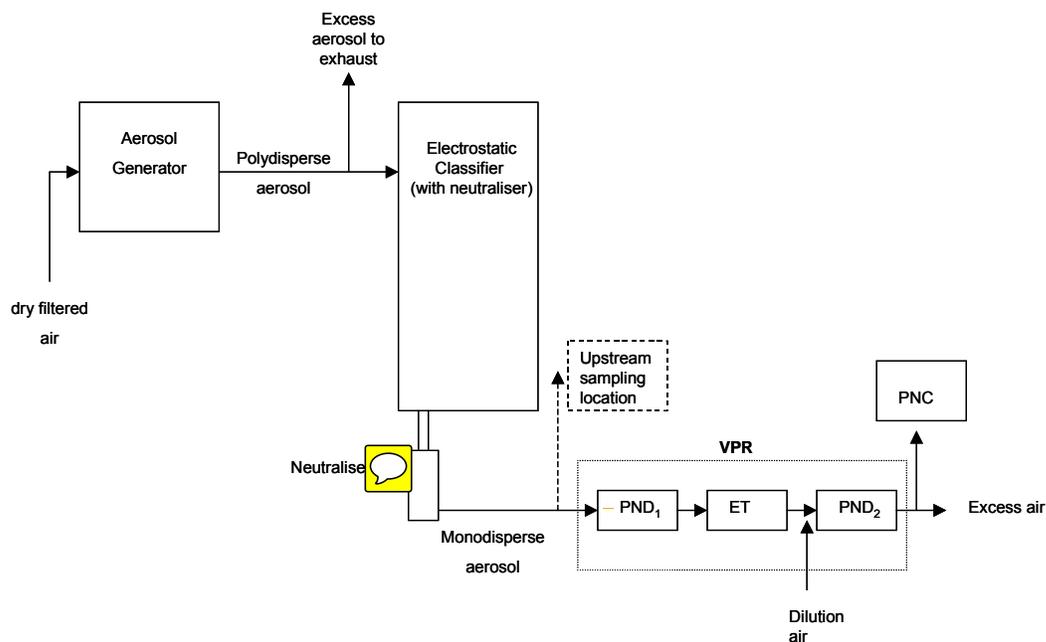
## 7 References

- <sup>1</sup> Amendments to UNECE Regulations, Regulation No. 83, Proposal for draft supplement 7 to the 05 series of amendments to Regulation No.83, ECE/TRANS/WP.29/GRPE/2007/8/Rev.1, <http://www.unece.org/trans/doc/2007/wp29grpe/ECE-TRANS-WP29-GRPE-2007-08r1e.pdf>
- <sup>2</sup>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
- <sup>3</sup>Fletcher, R et al Verification of a gas mask calibrant, NISTIR 7424 (*Aerosol generation using the electrospray*).
- <sup>4</sup>Jing, L Standard Combustion Aerosol Generator (SCAG) for Calibration purposes, 3<sup>rd</sup> ETH Workshop, 9-10 August 1999, [http://www.sootgenerator.com/documents/Pub-ETH-Workshop1999\\_Ji.pdf](http://www.sootgenerator.com/documents/Pub-ETH-Workshop1999_Ji.pdf)
- <sup>5</sup>Marshall, IA Particle Number Counters Calibration/Validation Procedures, report to Department for Transport, ED47382004/PNC, Revision 5, December 2007.
- <sup>6</sup>Mitchell, JP Aerosol Generation for Instrument Calibration, UKAEA, AEEW – R-2092
- <sup>7</sup>VDI Guideline 3491 Parts1 to 16, The Association of German Engineers, "Particulate matter measurement; production methods of test aerosols". <http://www.vdi.de/en/7636.0.html>. (*Generation of ultrafine particles: Parts 2 (foundations and synoptics), Part 3 (Generation of Latex aerosols using nozzle atomizers), Part 6 (Platinum oxide generator) and Part 16 (Generation of carbon aerosols using a spark aerosol generator)*).

## 8 Annex 1: example of primary calibration of VPR

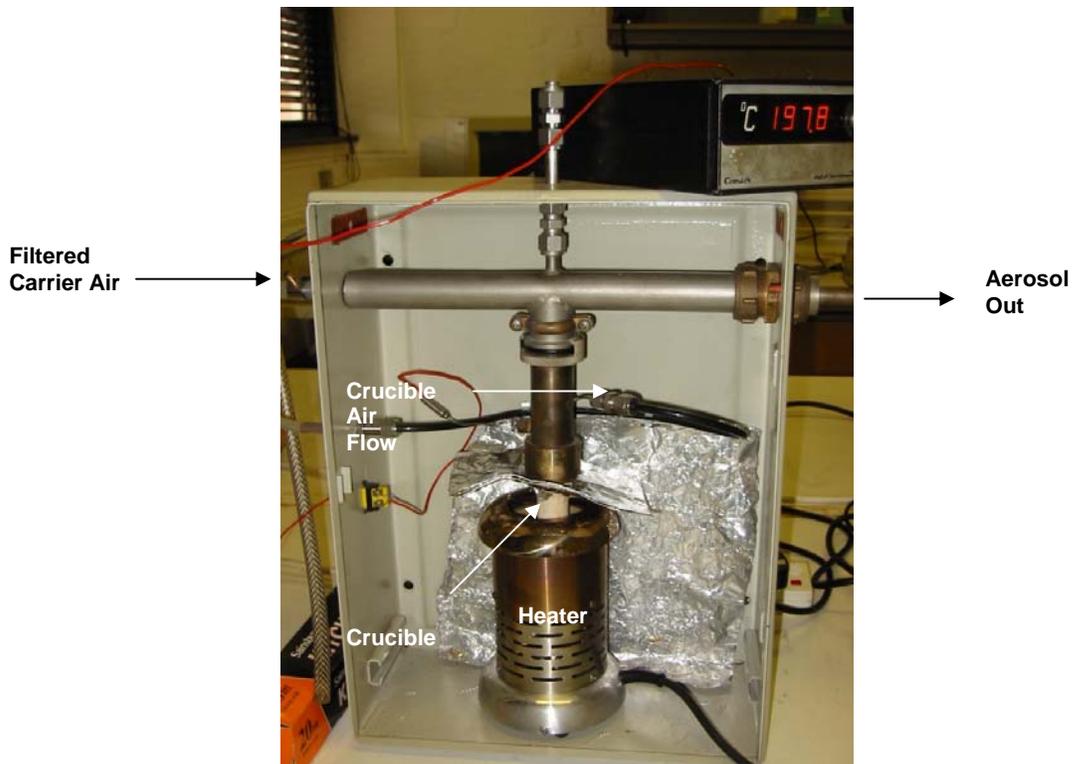
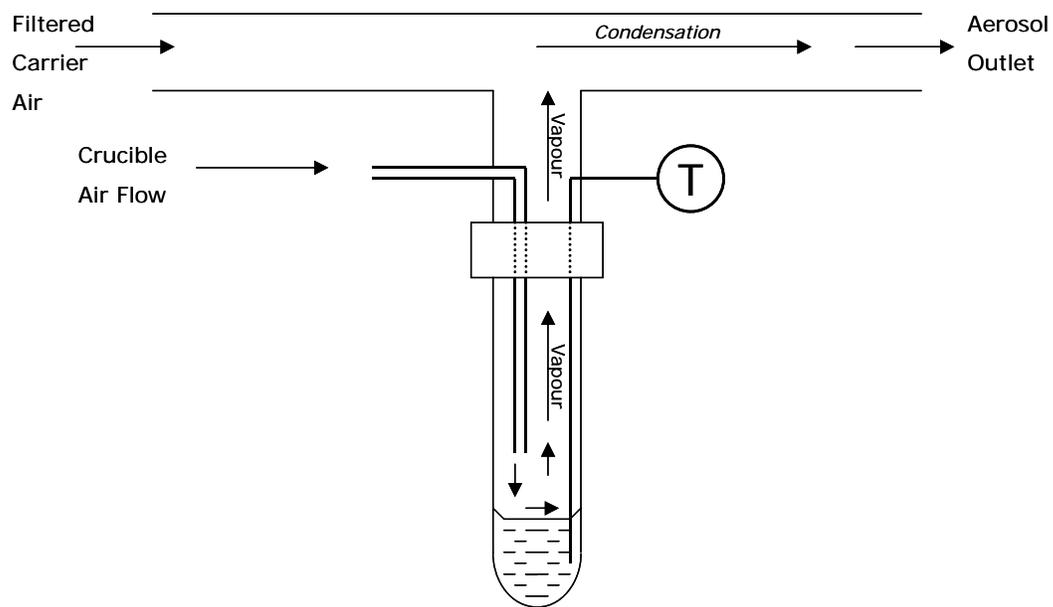
The following example describes one method for calibrating a VPR. The experimental set-up is illustrated in Figure 2.

Figure 2: Calibration of the VPR

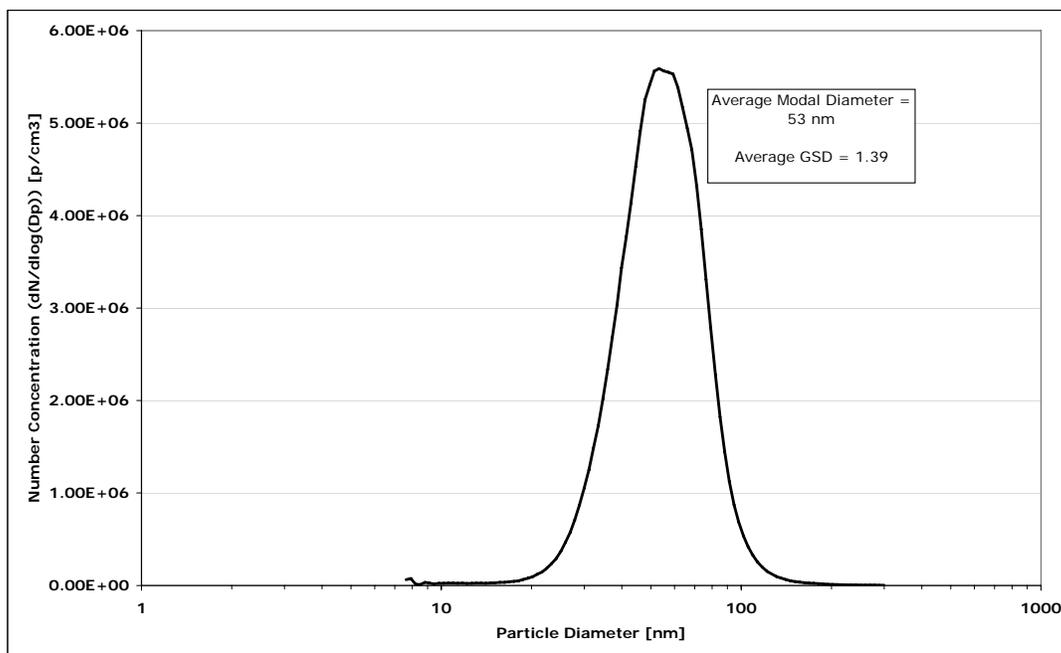


The aerosol generator consisted of a ceramic crucible heated via an electric Bunsen (Figure 3). The bulk material (sodium chloride for particle concentration reduction factor measurement and tetracontane for volatile particle removal efficiency) was placed in the ceramic crucible and heated to near its 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 sodium chloride aerosol is presented in Figure 4.

Figure 3: Condensation Aerosol Generator



**Figure 4** Typical size distribution of a polydisperse sodium chloride calibration aerosol



*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 (> 5000 particles cm<sup>-3</sup> for sodium chloride and >10,000 particles cm<sup>-3</sup> for tetracontane), reducing the GSD (geometric standard deviation) to around 1.2.

Two PNCs were used to simultaneously record the number concentrations upstream (at the inlet of PND<sub>1</sub>) and downstream (at the outlet of the VPR).

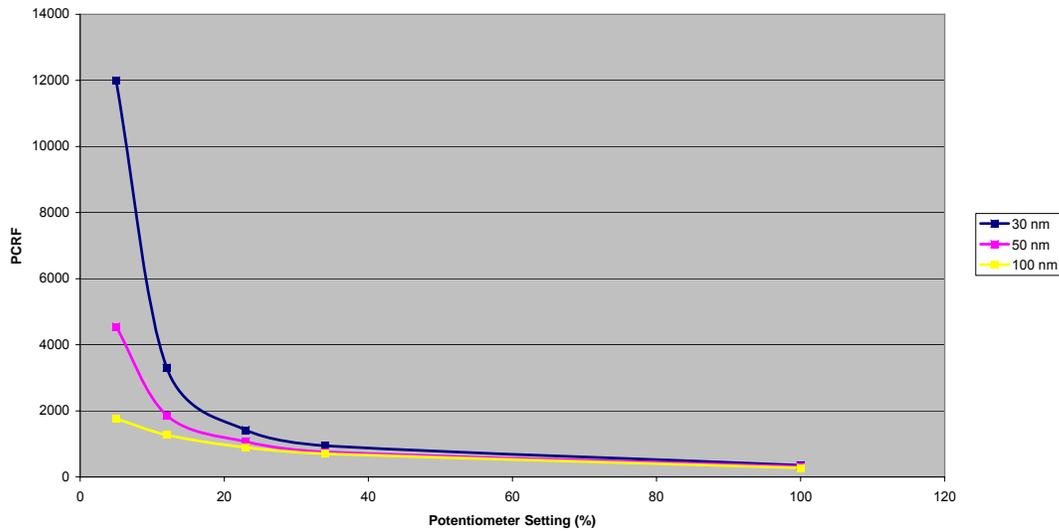
*Note: It is imperative that the response of these PNCs is verified before the VPR is calibrated; this is to ensure that the two PNCs are equivalent. The upstream and downstream concentrations are directly compared with each other to calculate the particle concentration reduction factor and volatile particle removal efficiency of the VPR; therefore the PNCs must be compared with each other. The secondary calibration method for PNC calibration is recommended, as detailed in the 'Condensation Particle Counter Calibration Procedures'<sup>5</sup>. The linearity of response in terms of the R<sup>2</sup> coefficient must be greater than 0.97, else the PNCs do not respond linearly over the measured concentration range. If the two PNCs do not meet these requirements the upstream and downstream concentrations may not be directly compared. The PNCs response at all particle sizes and concentration should be compared and measurements taken with one PNC adjusted to align with the other PNC. Additionally the data from the upstream and downstream PNCs must be time aligned to account for the VPR residence time.*

Additionally, care must be taken when comparing the results from two PNCs if they are not the same model, this was particularly important in this example as one PNC had been modified to provide lower size detection limits of  $23\pm 3$  and  $37\pm 4$  nm for 50 % detection efficiency ( $D_{50}$ ) and 90 % detection efficiency ( $D_{90}$ ) respectively than the standard model of TSI PNC 3010. The AEA PNC was unmodified having a  $D_{50}$  of  $9.5\pm 0.5$  nm and  $D_{90}$  of  $13.5\pm 0.5$  nm. Thus significant differences between the concentrations reported by both PNCs would be expected if the calibration particles were smaller than around 40 nm. For the VPR calibrations at 30 nm the AEA PNC 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 PNC 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 PNCs and the VPR inlet, thus ensuring any losses within the pipe work were the equivalent for all measurements.

For each monodisperse diameter and nominal dilution setting the system was allowed to stabilise for at least 1 minute before selecting the next setting. When the measurements were complete at each nominal dilution setting the particle diameter was changed. The results were analysed to calculate the particle concentration reduction factor at each nominal dilution setting (Figure 5).

**Figure 5: Example of measured particle concentration reduction factors for 30, 50 and 100 nm diameter particles at five dilution settings.**



*Note: Figure 5 is an example of the measured particle concentration reduction factors, the VPR under test required maintenance and would not have passed calibration.*

The measured upstream and downstream number concentrations of 30 nm tetracontane volatile particles are shown in Table 3. The final two columns list the measured downstream concentration after the particle concentration reduction factor has been applied to the data and compares this with the allowed downstream concentration (1 % of the upstream concentration). The measurement was repeated five times and the VPR met the criteria for all repeats.

**Table 3 Volatile Particle Removal Efficiency**

Repeat	Upstream Concentration (cm <sup>-3</sup> )	Downstream Concentration (cm <sup>-3</sup> )	PCRf	Adjusted Downstream Concentration (cm <sup>-3</sup> )	Allowed Downstream Concentration (cm <sup>-3</sup> )
1	11125	0.24	353	84.72	111.25
2	10736	0.24	353	84.72	107.36
3	12399	0.34	353	120.02	123.99
4	15472	0.43	353	151.79	154.72
5	15338	0.43	353	151.79	153.38



## 9 Annex 2: Example of particle concentration reduction factors ( $f_r$ )

Table 4 lists the minimum number of particle concentration reduction factors required for primary calibration for VPRs with a variable secondary diluter (PND<sub>2</sub>).ie all fifteen factors are required.

For VPRs with a fixed PND<sub>2</sub> dilution setting only the first set of particle concentration reduction factors would be required (highlighted in yellow).

The particle concentration reduction factors in Table 4 are shown as an example, as measured for the VPR calibrated in Annex 1.

**Table 4: Examples of particle concentration reduction factors ( $f_r$ )**

Primary Diluter Setting (PND <sub>1</sub> )	Secondary Diluter Setting (PND <sub>2</sub> )	Particle concentration reduction factor ( $f_r$ )
1	1	313
2	1	802
3	1	1128
4	1	2147
5	1	6104
1	2	-
2	2	-
3	2	-
4	2	-
5	2	-
1	3	-
2	3	-
3	3	-
4	3	-
5	3	-



## 10 Glossary of Terms

Term	Acronym	Definition
Diesel Particulate Filter	DPF	A particle filter that is used within the exhaust system of a diesel vehicle.
Dilution Factor	DF	A measure of the degree of dilution achieved by a diluter operating at specified conditions.
Downstream		Refers to the sampling location at the outlet of the VPR <i>i.e.</i> after the second diluter PND <sub>2</sub>
Electrostatic Classifier		Primary-standard instruments that produce highly monodisperse, submicrometer aerosols.
Evaporation Tube	ET	Heated tube used to vaporise volatile particles from the aerosol sample.
Gas Concentration Reduction Factor		A factor used in an optional quality control check of the VPR calculated from the measured dilution of a certified gas concentration.
High Efficiency Particulate Air Filter	HEPA	An air filter capable of removing particles at a high efficiency as specified in EN1822:1998.
Particle Concentration Reduction Factor (fr)	PCRF	A factor used to describe the performance of the VPR. The PCRF incorporates actual dilution and all particle losses within the VPR. PCRF is calculated from: $f_r(d_i) = \frac{N_{in}(d_i)}{N_{out}(d_i)} \quad (12)$ Where $N_{in}(d_i)$ = upstream particle number concentration for particles of diameter $d_i$ ; and $N_{out}(d_i)$ = downstream particle number concentration for particles of diameter $d_i$
Particle Number Counter	PNC	An instrument used to count particle number concentration.
Particle Number Diluter	PND	Instrument used to dilute the number of particles in the sample.
Upstream		Refers to the sampling location at the inlet of the VPR <i>i.e.</i> PND <sub>1</sub> inlet
Volatile Particle Removal Efficiency		A measure of the VPR's ability to vaporise volatile particles.
Volatile Particle Remover	VPR	Instrument capable of diluting the sample in one or more stages to achieve a particle number concentration below the upper threshold of the single particle count mode of the PNC and to achieve a gas temperature below 35 °C at the inlet to the PNC while suppressing nucleation within the sample.



 **AEA Energy & Environment**  
From the AEA group

Harwell International Business Centre  
Didcot  
Oxfordshire  
OX11 0QR

Tel: 0845 345 3302  
Fax: 0870 190 6318

E-mail: [info@aeat.co.uk](mailto:info@aeat.co.uk)

**[www.aea-energy-and-environment.co.uk](http://www.aea-energy-and-environment.co.uk)**