Calibration of DMS Series Fast Particulate Spectrometers

Contents

Introduction

This document outlines how a DMS500 or DMS50 is currently calibrated for particle size and number. The fundamental base calibration arises from a detailed computer model of the instrument, which takes into account the aerosol and sheath flows, the unipolar diffusion charger characteristics, and classifier column voltage, pressure, temperature and geometry. The probability distribution of electrical charges on a particle of given size is calculated, and this is used to predict which rings such particles are likely to land on, given the conditions of the classifier. This model produces a complete *transfer function* for the instrument, mapping particle size to ring currents (Figure 1).
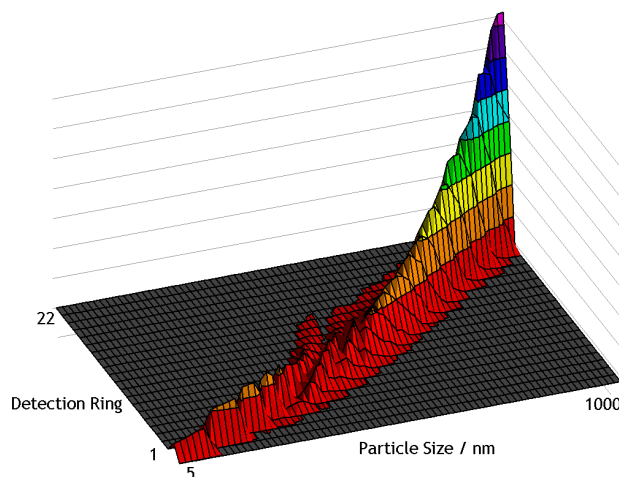

Figure 1: DMS500 Transfer Function

After the DMS has been checked for correct operation, with its internal flows within tolerance, the computer generated transfer function is empirically adjusted with real

aerosols. The lognormal data parameterisation (user interface 2.01 et seq.) is used for this (as opposed to the discrete spectrum) as it provides a direct reading of the mean diameter of individual aerosol modes, and improves the spectral resolution.

Sub-100 nm calibration for size and number

This procedure is closely related to the calibration procedure generally used by CPC manufacturers[†], using a standard electrometer, whilst taking into account the size information provided by the DMA. Number calibration against a CPC is not performed for the reasons discussed in Appendix A.

The aerosols used are:

- Nucleated Liquid Sulphuric Acid (~ 15nm). See appendix B for the generation method.
- Atomised and dried solid Sodium Chloride (~ 50 & 100 nm)

These aerosols are size selected with a TSI 3080 electrostatic classifier (with 3081 DMA). The monodisperse aerosol stream is split into two, with half passing through a radioactive neutraliser into the DMS, and the other half passing through a Faraday cage electrometer system. The size and concentration are measured by the DMS, and compared with the size set by the DMA and the concentration measured by the electrometer. The setup is shown in Figure 2.
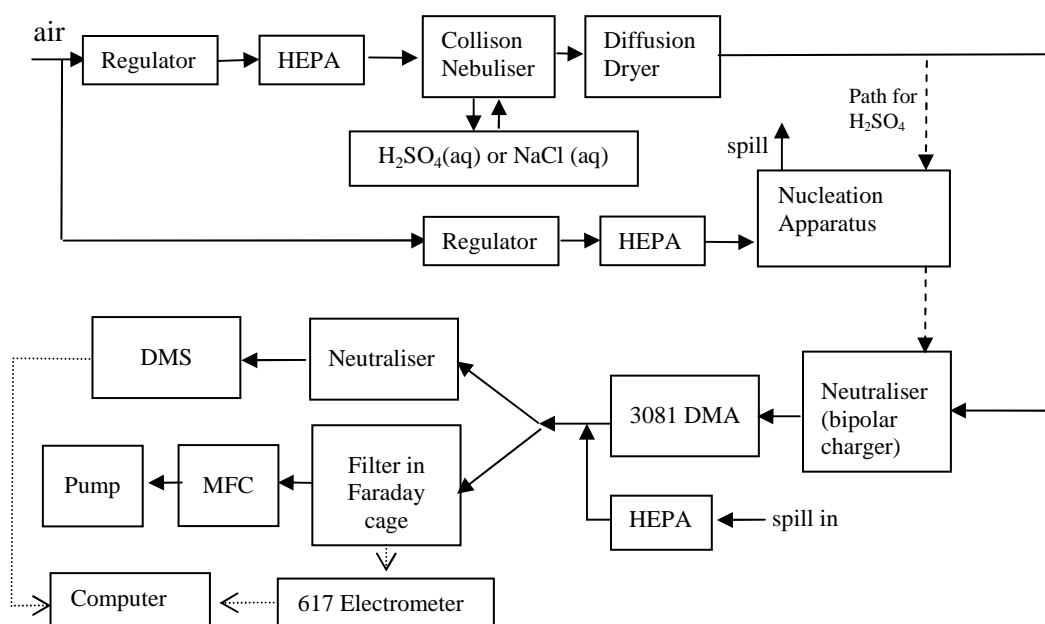

Figure 2: Sub-100 nm Calibration

The electrometer system is based around a HEPA filter enclosed by a conductive shell. By Gauss' theorem, charged particles arriving within the shell (i.e. collected by

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[†] e.g. Liu, B.Y.H and Pui, D.Y.H. (1974) A Submicron Aerosol Standard and the Primary Absolute Calibration of the Condensation Nuclei Counter, *J. Colloid Interface Science*, **47**, 155.

the filter) will induce a charge in the shell, which will produce a measurable current when connected to a sensitive electrometer (Keithley model 617). A pump draws a comparable flow to that of the DMS through the filter, metered and controlled by an Aalborg mass flow controller. The number concentration (in cm^{-3}) is then given by:

$$N = \frac{I}{eQ}$$

where Q is the flow through the filter (in cm^3 s^{-1}), e is the electronic charge (in C) and I is the measured current (in A).

This calculation assumes that the aerosol leaving the DMA is singly charged. In practice this is achieved by ensuring that the original aerosol has very few particles larger than the required DMA size, as these particles might be doubly charged yet still appear to have the same electrical mobility as singly charged particles of the required size. This is ensured by (a) the H_2SO_4 aerosol from the nucleation apparatus being essentially monodisperse and (b) carefully choosing the NaCl(aq) concentration so that DMA size selection occurs from the largest particle end of the polydisperse aerosol distribution.

Supra-100 nm calibration for size

NIST Traceable Polystyrene Latex (PSL) spheres of certified size are used. These are nebulised in aqueous suspension, then dried and neutralised before being sampled by the DMS. Particles produced by the nebuliser which have been dried will be very highly charged, so they must be neutralised before DMS measurement, or both measured size and number will be affected. The hardware setup is shown in Figure 3.
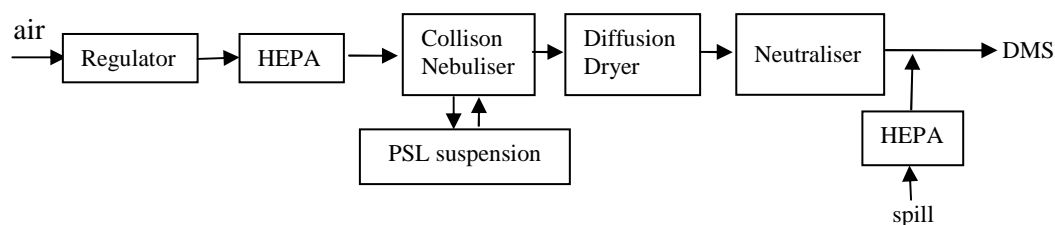

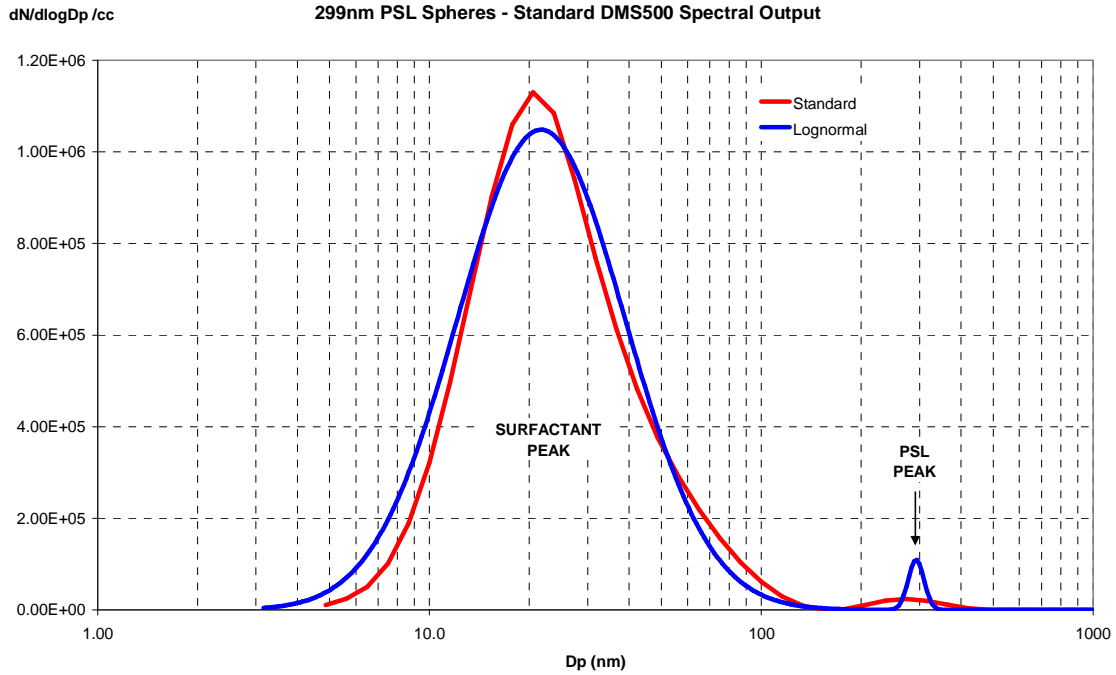
Figure 3: PSL Aerosol Generation

Figure 4: 299 nm PSL sampled with the DMS500

Figure 4 shows an example of a PSL spectrum. The lognormal data inversion gives a much higher spectral resolution than the discrete spectrum. Note the broad mode at small sizes, caused by surfactants and impurities. PSL is unsuitable for use below 100 nm, as this "surfactant mode" can mask or distort the PSL peak for smaller PSL sizes. Also, it important to dilute the PSL suspension sufficiently so that no more than one PSL particle is present per water droplet, or particle aggregation can occur upon drying, giving an inaccurate mobility size. This problem is less significant for larger PSL sizes.

The PSL point at 100 nm provides a useful cross-check of the calibration of the DMA used for the sub-100 nm region. Due to the issue of multiple charging, it is not yet practical to provide number certification for larger particle sizes, so the original computer model is currently relied upon here.

Certification

After empirical adjustment of the transfer function, a new .dmd file containing the new matrix is loaded, and the instrument is then checked against similar aerosols. Certification is dependent upon:
- The size being within 5% of the standard in the region of 5–300 nm.
- The size being within 10% of the standard for diameters > 300 nm.
- The number concentration being within 10% of the standard.

Agglomerate Aerosols: Calibration for Diesel

Because highly fractal aerosols acquire more charge in this type of instrument, a separate calibration is provided for use with Diesel engines[†]. In particular, this ensures greater accuracy when compared with a Particle Measurement Programme (PMP) number measurement system. The Diesel .dmd calibration file supplied with each DMS uses this calibration for the accumulation mode of the lognormal output only; the nucleation mode and the continuous "red" spectrum use the standard calibration and therefore one can expect the accumulation mode lognormal peak to indicate a lower concentration and larger size than the continuous spectrum.

The calibration procedure is similar to the sub-100nm calibration for size and number descried above, except rather than being NaCl or H_2SO_4 the aerosol source is soot from a propane fuelled CAST device. The soot is pre size-selected with a second DMA to be monodisperse thus reducing the likelihood of multiple charging affecting the gain calibration. Certification by this process is at 50, 75, 100 and 200 nm. The instrument is expected to meet the standard within 10% for both size and number.

To allow for the same level of accuracy in mass calculation as was achieved in Symonds et al. (2007)[‡], since the introduction of the agglomerate calibration, the diameter to mass rule has been revised for this calibration to be Mass = $2.2 \times 10^{-15} \times D_p^{2.65}$, and the Diesel .dmd file now uses this rule.

The GDi .dmd file uses the standard "spherical" calibration for both nucleation and accumulation modes as this gives the best results for particle number and mass, see application note DMS08 for more details.

Calibration for 2.5 µm and switchable instruments

This is similar to the standard spherical calibration, except that monodisperse DEHS (oil) aerosol from a Sinclair-LeMer type condensation aerosol generator is used for DMA based size/number calibration between 100 and 500 nm, and PSL spheres are used beyond this size.

Appendix A: Reasons For Not Using a CPC.

The practical minimum concentration for such a measurement with a DMS type instrument ($> 10^4$ cm^{-3}) would mean that most CPCs would be operating in *photometric mode*, (as opposed to direct count mode), which is based upon bulk optical properties of the aerosol and is dependent upon manufacturer calibration. It is

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[†] Symonds, J.P.R., & Reavell, K.St.J. (2007). *Calibration of a Differential Mobility Spectrometer*, European Aerosol Conference, Salzburg, **T02A034**

[‡] Symonds, J.P.R., Reavell, K.St.J., Olfert, J.S., Campbell, B.W., & Swift, S.J. (2007). *Diesel Soot Mass Calculation In Real-time With A Differential Mobility Spectrometer*, Journal of Aerosol Science, **38** 52–68

this mode which CPC manufacturers calibrate against an electrometer as the primary standard, so the use of a CPC would be a secondary standard in this case. Figure 5 shows a comparison of the DMS500 with a TSI 3022 CPC. A sodium chloride aerosol with a GMD of ~ 55 nm and a GSD of ~ 1.9 was used. The DMS500 and CPC were fed with the same aerosol, and a DMS500 analogue input was used to log the CPC. Large scale concentration fluctuations in the aerosol are due to deliberate changes in the dilution flow, small scale fluctuations are due to the nebuliser's air regulator. It can clearly be seen from Figure 5 that the DMS500 and CPC are in excellent agreement on total number whilst the CPC is in count mode, but as soon as the CPC enters photometric mode an offset is seen on the CPC's output.

However, these data are rather close to the noise floor of a DMS, and so to use a CPC in count mode for certification would require an additional diluter, which in itself would need calibration and would be an additional source of error. An SMPS (DMA + CPC) is a complex system, reliant upon its own calibration and complex data inversion, so this is not used either.
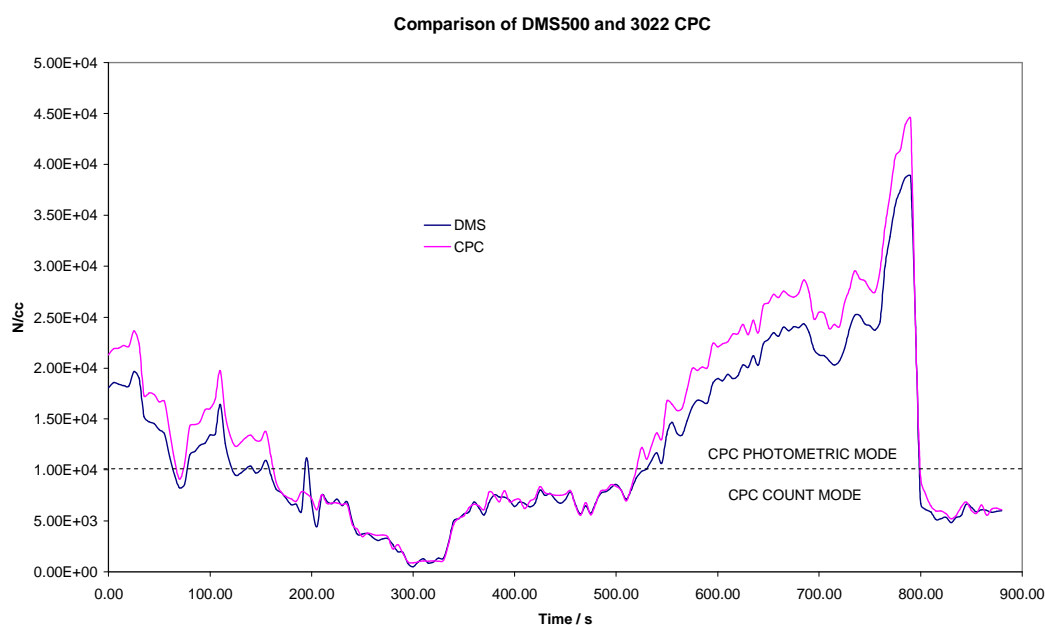

Figure 5: Comparison of DMS500 and 3022 CPC

Appendix B: Sulphuric Acid Aerosol Generation.

Sulphuric acid is nebulised and then partially dried before passing into the nucleation apparatus. This consists of a heated pipe (~ 120 °C) to vaporise some of the nebulised H_2SO_4, followed by a cold pipe into which dilution air is added to cause re-nucleation of H_2SO_4 and hence the generation of very small particles. This aerosol has two characteristic peaks, the nucleation mode at < 20 nm and the remaining un-evaporated acid mist at >20 nm. The apparatus can be tuned in size and in concentration ratio of the two peaks by adjusting the temperature and flows; this is to ensure the >20 nm peak is negligible in concentration compared with the nucleation peak and that the nucleation peak is smaller than 15 nm in size for selection by the DMA. For more details on this method of aerosol generation, see Cambustion Application Note DMS02.