# Aerosol laboratory assessment of particle sizing instruments for vehicle emissions

**Emma Payne, netcen , AEA Technology plc.**

This project was designed to assess the suitability of a number of candidate instruments for measuring vehicle emissions. This phase of the project had two main objectives, *viz.*, to develop an easily controlled aerosol generator of stable output which was capable of producing a wide range of particle diameters and concentrations, and subsequently, to use the aerosol generator to characterise the performance of a range of particle measuring instruments. This characterisation was to be in terms of repeatability, linearity, detection efficiency and particle sizing capability.

The test aerosol was generated from a solution of sodium chloride in a 50:50 mixture of methanol and water, by means of an ultrasonic nebuliser. The ultrasonic nebuliser used a piezoelectric crystal to generate ultrasonic waves near the surface of the solution. This intense energy caused the wave to break producing a dense aerosol of single droplets of solution. The droplets were subsequently dried to allow the sodium chloride within each solution droplet to crystallise into a solid particle. The frequency of the crystal was controlled to produce a range of droplet concentrations and diameters. In addition, feed solutions of various sodium chloride concentrations could be utilised to produce a wider range of modal particle diameters, as these were dependent on the quantity of sodium chloride present in each droplet. This technique produced aerosol concentrations of the order of $1 \times 10^5$ # $cm^{-3}$. A high aerosol concentration was an important factor as the aerosol was to be classified electrostatically and this can reduce particle concentrations by up to two orders of magnitude. Another key feature of the generator was it had to be capable of producing a stable output over a time period sufficient to assess an instrument.

The droplets were generated by the nebuliser directly into a large glass bell jar wrapped in heating tape and lined with silica gel desiccant. This heated drying chamber was used to evaporate solvent from the aerosol droplets and absorb the resulting vapour into the desiccant to prevent subsequent recondensation onto the solid sodium chloride particles. Considerable time was spent ensuring that the residence time of the aerosol within the chamber was sufficient to dry the particles without their forming agglomerates. There was a finite time limit of around 30 minutes for aerosol generation before the desiccant became saturated. This was judged sufficient time to undertake an instrument assessment. An additional check to confirm that the aerosol was dry was performed using a transparent silicon "U" tube fitted at the outlet of the drying chamber. This enabled a visual check for water condensing in the tube and collecting in the "U" tube. The dry solid particles were electrostatically classified to produce a monodisperse aerosol. The particle diameter of interest could be selected by fixing the voltage of the central rod of the classifier. This enabled a range of monodisperse aerosols to be generated. Each of the five test aerosols generated (with nominal particle diameters of 30, 60, 120, 200 and 400 nm) were present in concentrations of around 10 particles per second. The graph shown in slide 6 shows the narrow size distributions for each aerosol generated, confirming that each distribution is extremely monodisperse with geometric standard deviations less than .2.

Aerosol concentration was varied by introducing a diluter at the outlet of the electrostatic classifier. The venturi diluter was used to produce dilution ratios between 300 and 10,000 times. The diluter was well characterised having been previously calibrated with a carbon monoxide gas standard. It enabled either the monodisperse or polydisperse aerosol concentrations to be varied whilst delivering a positive pressure sample to the instruments under assessment. This diluter had the advantages of removing the pressure fluctuations in the system but also maintaining a high sample flow rate to suit a wide range of aerosol measuring instruments.

By way of an example, the aerosol concentration was varied using the venturi diluter, giving dilution ratios of between 300 and 9000 times for one feed solution. Slide 8 shows the SMPS-measured particle diameter distributions for each of the selected dilution ratios.

The modal size of each aerosol concentration was 45 nm irrespective of the value of the dilution ratio, giving confidence that the diluter did not significantly alter the size distribution.  The overlap of the two scans (solid and dotted lines) for each dilution ratio also gave confidence that the generated aerosol was sufficiently stable to undertake repeatability assessments.  It should be noted that these data was obtained using the polydisperse aerosol, *i.e.* without any electrostatic classification.

The particle size distributions of the calibration aerosol demonstrated that the aerosol generator was able to produce a dry, polydisperse unimodal aerosol at 45 nm and at controllable concentrations.  This enabled the linearity of aerosol instrument response to changing concentration and instrument detection efficiency as a function of particle diameter to be determined.  Slide 10 is an example of the linearity determination of a particle counting instrument.  The challenge aerosol was polydisperse with a modal diameter of 45 nm.  The diluter was used to control the aerosol concentration that is measured by the particle counting instrument under assessment.  A high dilution ratio corresponds to a low aerosol concentration, but the graph is plotted at 1/dilution to produce a positive gradient which passes through the origin.  This instrument had a very good linear response across all aerosol concentrations measured.

A "reference" Scanning Mobility Particle Sizer (SMPS) was added to the original aerosol generator to allow direct comparisons to be made between the reference instrument and the instrument under assessment.  The reference SMPS had been rigorously calibrated in terms of its flow rates, delay times, particle losses and linearity.  The counting efficiency of an instrument could be assessed by simultaneously sampling the calibration aerosol with the instrument and the reference SMPS.  The CPC Sensitivity table contains the nominal particle diameter generated by the classifier and the actual particle diameter measured by the reference SMPS, as well as the concentration measured by the SMPS and CPC.  Very good agreement was achieved between the measured concentrations for the particle diameters generated.  This is illustrated more clearly in the following graph of CPC sensitivity.  CPC-measured concentrations were plotted as a percentage of the SMPS-measured concentrations to illustrate CPC sensitivity as a function of particle diameter. There was a uniform response close to 100% in the particle diameter range from 30 and 400 nm.  We can measure the sensitivity of the CPC in terms of its particle diameter compared to that measured by the SMPS.

An aerosol generator capable of producing a range of particle diameters between 30 and 400 nm at concentrations up to $1 \times 10^5$ # $cm^{-3}$ was successfully developed.  Aerosol concentration was readily controlled by means of a calibrated venturi diluter or by varying the concentration of the feed solution.

A dry aerosol was generated that was stable for up to 30 minutes before desiccant saturation became an issue.  The aerosol generator facilitated the characterisation of a number of aerosol measuring instruments in terms of linearity of response and detection efficiency.

Acknowledgements:

Dr I A Marshall, netcen

# Aerosol laboratory assessment of particle sizing instruments for vehicle emissions

Emma Payne

netcen
Aerosol Science Centre

6th International ETH-Conference on Nanoparticle Measurement
19th-21st August 2002

netcen

# Outline:

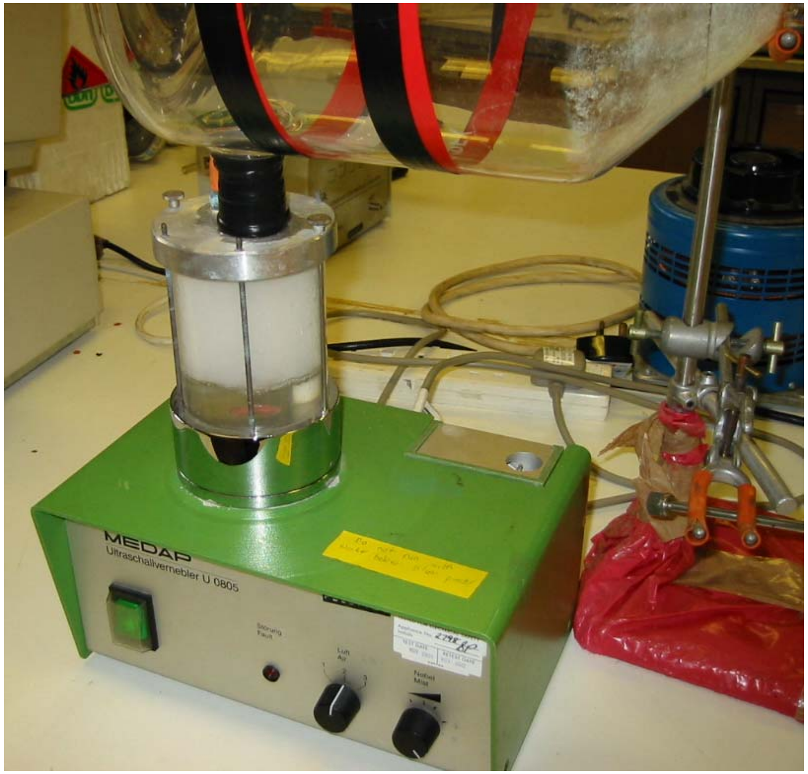
- Objectives
- Aerosol generator development
- Demonstration experiments
- Conclusions

# Project objectives:

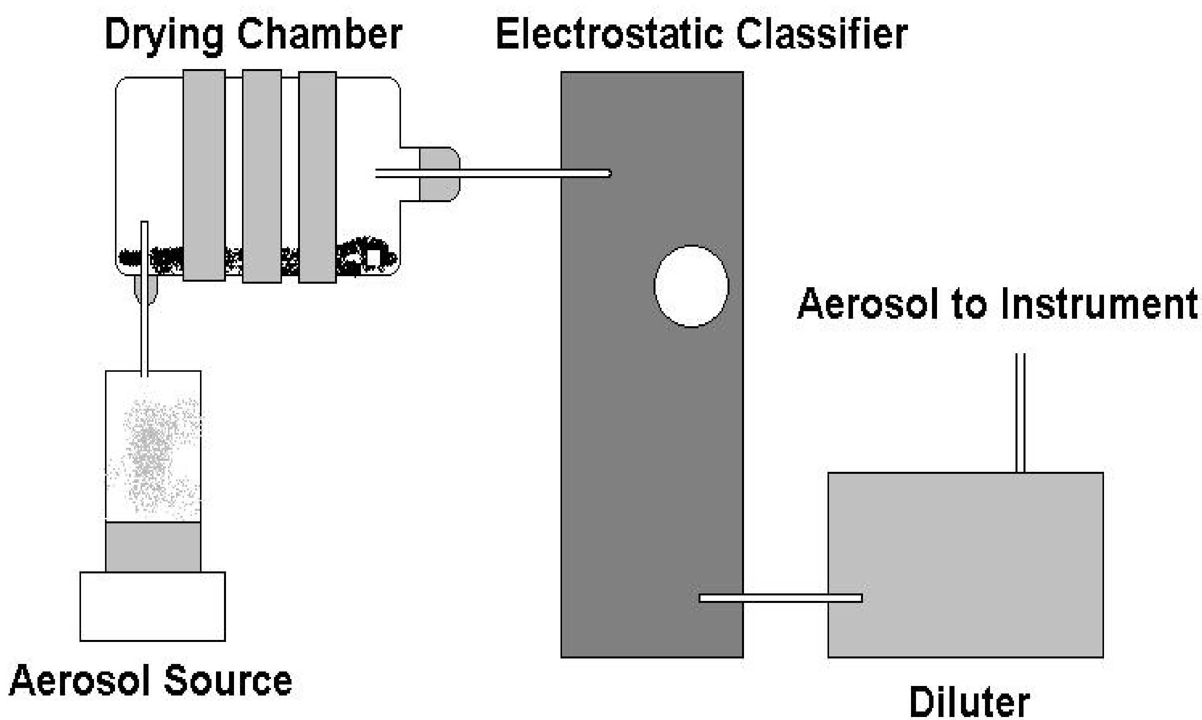
- To develop an aerosol generation facility having the following features:
  - Ultrafine monodisperse particles
  - Easily controlled particle diameter
  - Easily controlled particle concentration
  - Stable output
- To demonstrate the feasibility of using such a generator to characterise the performance of a range of particle measuring instruments with respect to:
  - Repeatability
  - Linearity
  - Detection Efficiency
  - Particle sizing capability

# Aerosol Generation

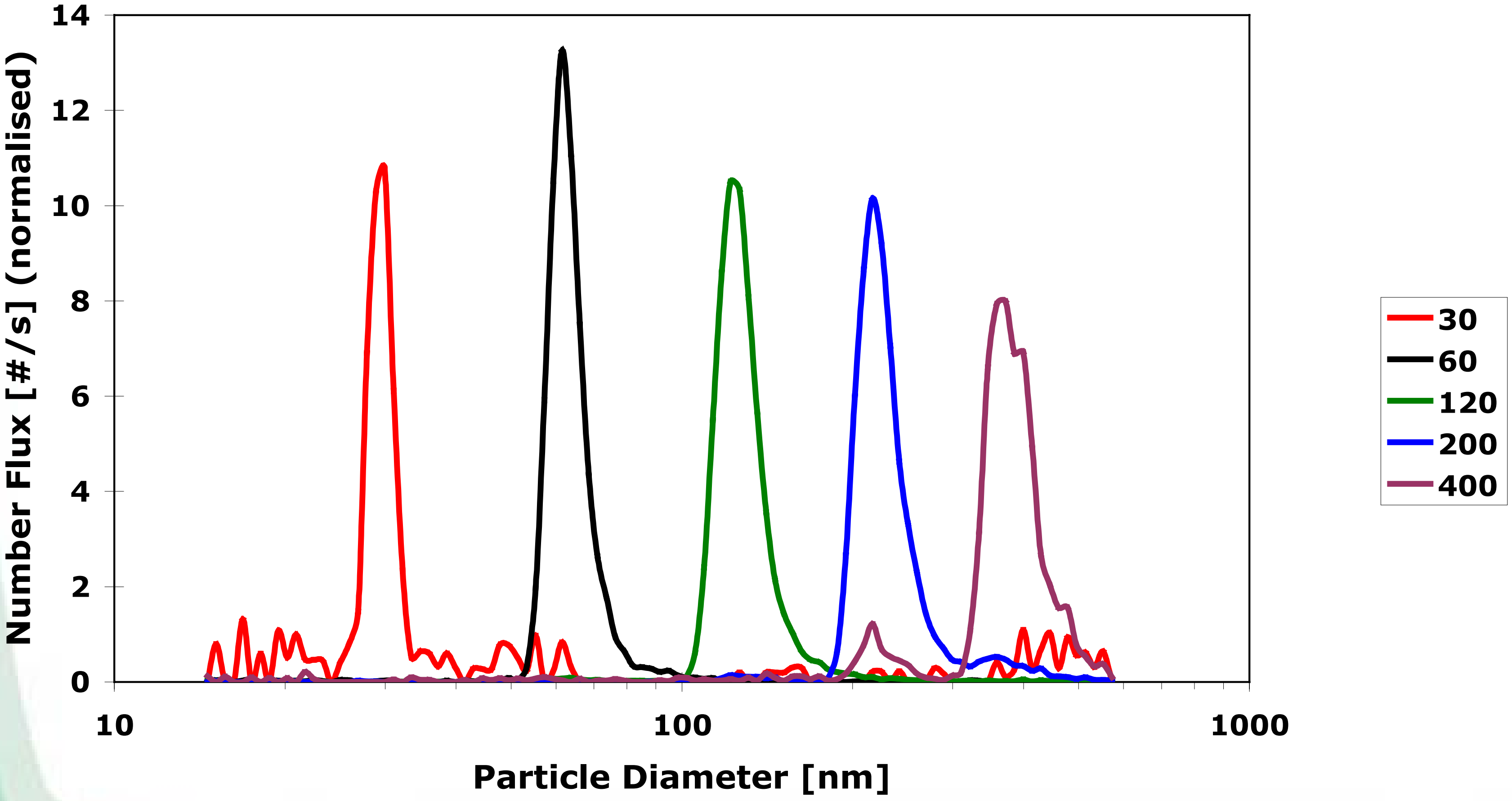
- Ultrasonic nebulisation of sodium chloride solution (in 50:50 water/methanol mixture)
- Range of particle diameters
- High concentration
- Stable



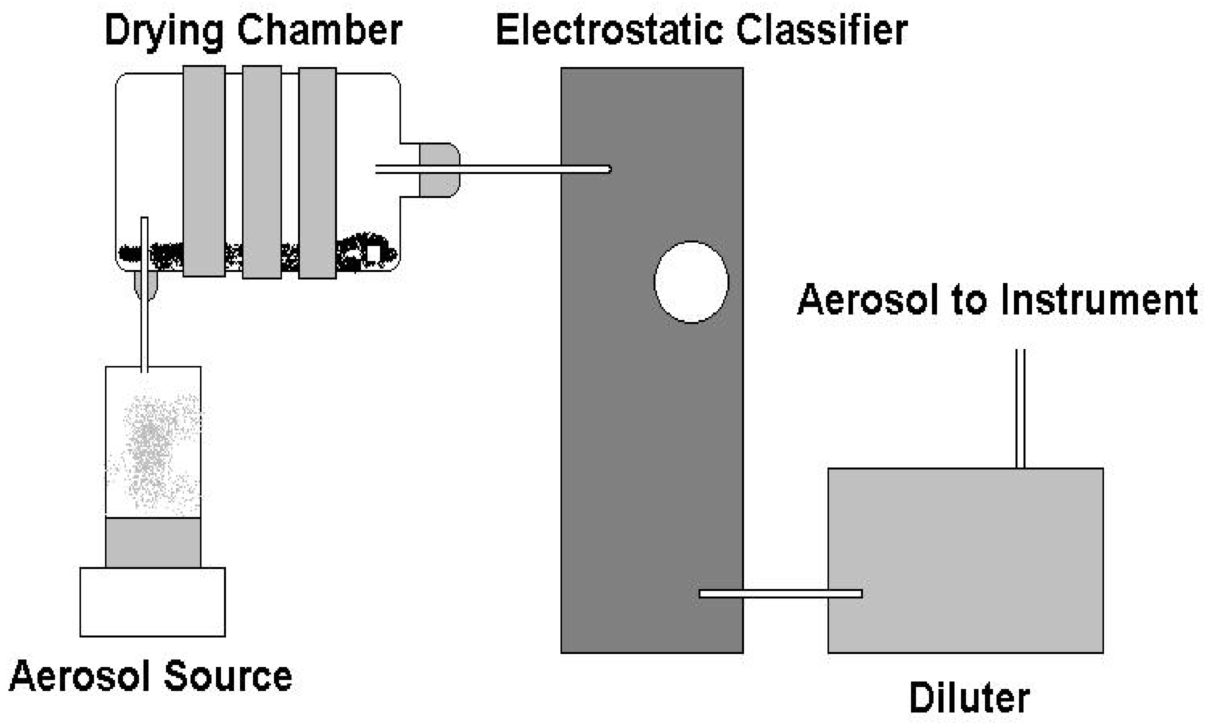
# Test Facility



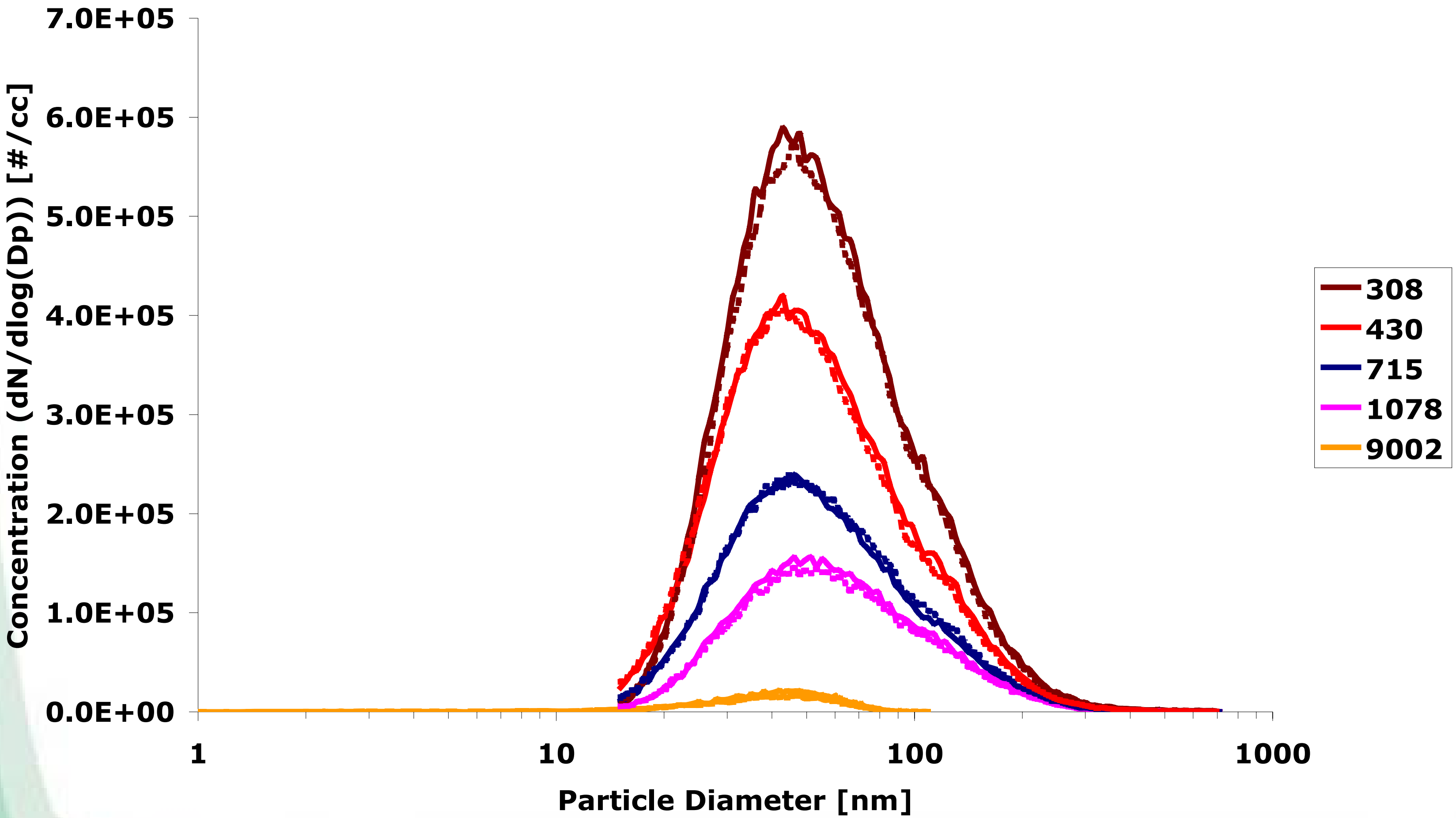
# Monodisperse Test Aerosols



# Test Facility



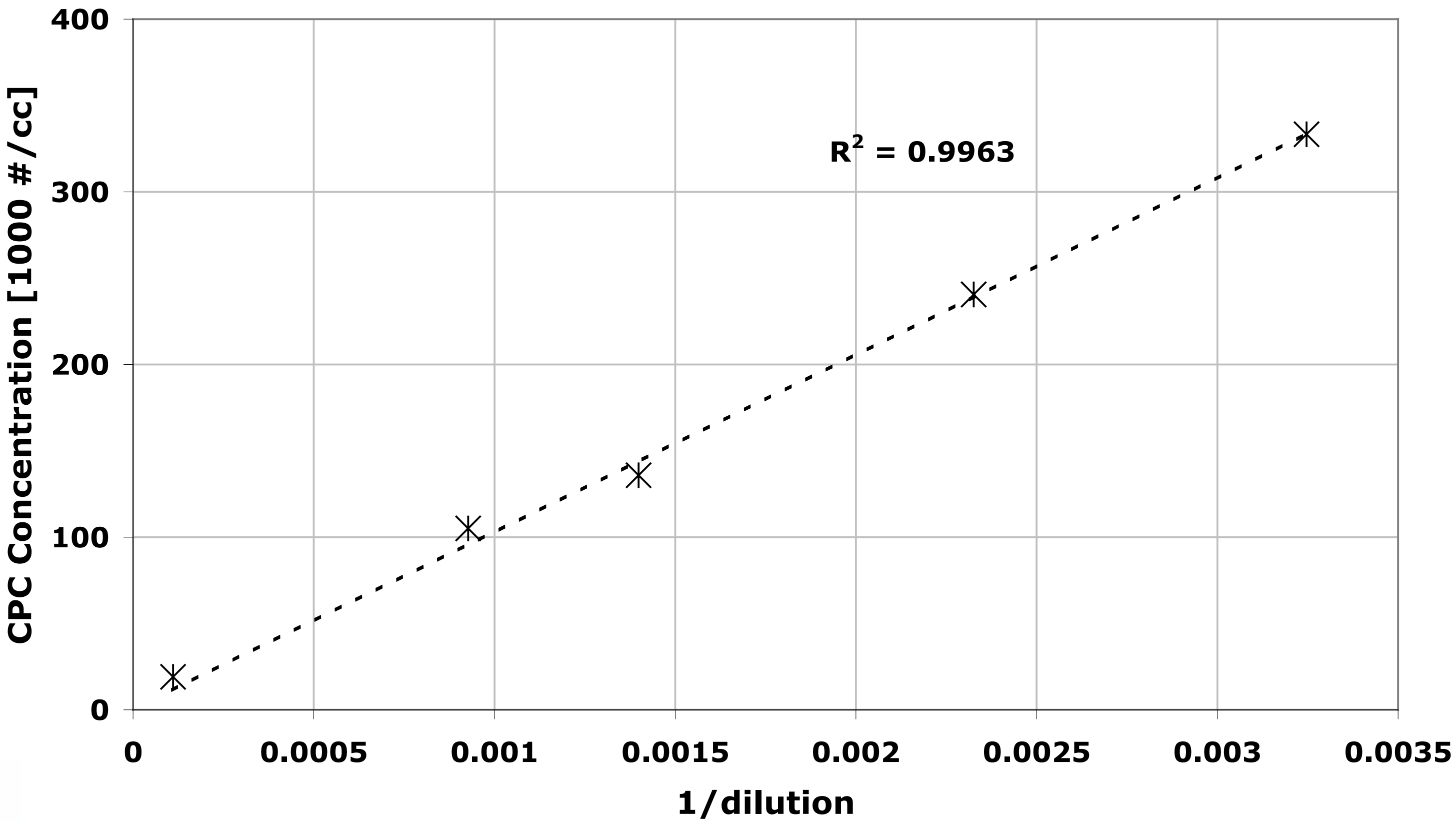
# Particle size distributions of calibration aerosol



# Applications:

- Linearity
- Detection Efficiency

# Linearity determination



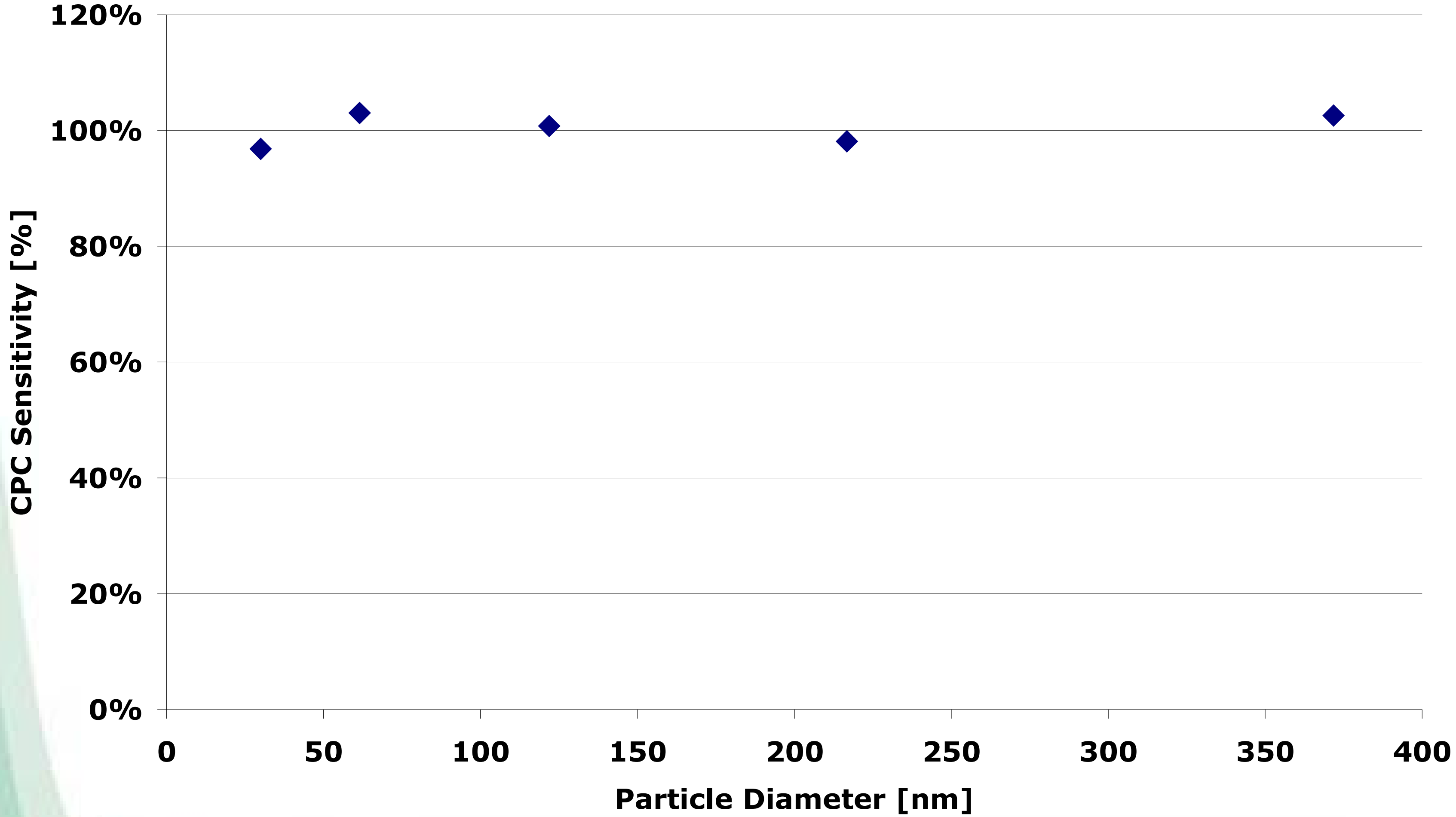
# Detection Efficiency

Polydisperse
aerosol

# CPC Sensitivity

| Nominal Particle Diameter (nm) | SMPS Modal Particle Diameter (nm) | SMPS Concentration (#/cc) | CPC Concentration (#/cc) |
|---|---|---|---|
| 30 | 29 | 1.42E+02 | 1.37E+02 |
| 60 | 61 | 3.06E+03 | 3.15E+03 |
| 120 | 121 | 9.27E+03 | 9.35E+03 |
| 200 | 216 | 6.22E+03 | 6.10E+03 |
| 400 | 371 | 9.75E+02 | 1.00E+03 |

# CPC Sensitivity



# Conclusions

A calibration aerosol generator has been developed:

- ✓ Monodisperse particles from 30 to 400 nm
- ✓ Diameter controlled via feed solution concentration
- ✓ Concentration controlled using a calibrated diluter
- ✓ Stable for up to 30 minutes

The feasibility of using the generator to characterise measuring instrument performance has been proven in a number of demonstration experiments.