

**REVIEW OF DIESEL PARTICULATE MATTER SAMPLING METHODS**

**Supplemental Report # 1**

**DIESEL EXHAUST PARTICLE MEASUREMENT INSTRUMENTS**

Prepared by

David B. Kittelson, Ph.D., Winthrop F. Watts, Jr., Ph.D. and  
Megan Arnold

University of Minnesota  
Department of Mechanical Engineering  
Center for Diesel Research  
Minneapolis, MN

July 31, 1998

## TABLE OF CONTENTS

	Page
Abstract.....	3
Introduction.....	3
Diesel aerosol size instrumentation.....	4
Inertial mobility instruments.....	4
Electric mobility instruments.....	5
Photoemission instruments.....	9
Mass spectrometry instruments.....	10
Radioactive tracer instruments.....	12
Other sizing instruments.....	12
GM and Ford lab visits.....	14
References.....	14
Appendix 1 - Lab survey summary.....	21

## ABSTRACT

This report describes instruments used to characterize diesel aerosol, summarizes information obtained from engine laboratory visits and present results from a diesel aerosol sampling questionnaire circulated to major engine labs. It is the first supplemental report to the final project report prepared for the Environmental Protection Agency (EPA) grant titled "Review of Diesel Particulate Matter Sampling Methods". This report is a revised version of a previous project report and is an accompaniment to Supplemental Report No. 2, titled "Aerosol Dynamics and Laboratory and On-Road Studies" (Kittelson, *et al.*, 1998a) and the final project report (Kittelson, *et al.*, 1998b).

## INTRODUCTION

Particle size distribution analysis of diesel aerosol can be divided into two principle tasks; sample collection and sample analysis. The size distribution of the aerosol is a function of the chemical and physical processes occurring between the point of aerosol generation and the point of aerosol collection. These processes, such as dilution, coagulation, condensation, homogeneous and heterogeneous nucleation, simultaneously affect the aerosol size distribution and are not well understood and are discussed in the second supplemental report. This report focuses on summarizing information on instruments used for the particle size analysis of diesel aerosol.

Instrumentation used for diesel aerosol size characterization is of essentially two types; those that provide size distribution data in real or near real-time and those that collect samples for subsequent analysis such as gravimetric, chemical or microscopic analysis. In many instances, the capabilities of these instruments are the limiting factor in how aerosol is sampled. The analytical methods used to characterize the diesel aerosol chemically and biologically are not covered in this report.

Aerosol sizing instruments may be divided into categories based on the measurement principle. Several issues pertain to the use of these instruments. Each instrument measures some property of the aerosol that relates to size, such as electrical mobility, aerodynamic diameter, optical behavior, inertial mobility, photoelectric emission, mass spectroscopy, etc. Each of these properties is related through analysis to aerosol parameters such as number, surface area, volume or mass-weighted size distribution. The investigator using these instruments should be aware of issues of relating the property being measured and the final output of the instrument. Because the instruments are based on different particulate properties, results from these instruments may not always be compared directly. In general, it is not possible to obtain particle size information on the entire five-decade size range from 0.001 - 100  $\mu\text{m}$  with a single instrument (Baron and Willeke, 1993). In most instances multiple instruments using different principles of operation are required to measure the diesel aerosol size distribution. Finally, many instruments are calibrated using a precisely controlled aerosol that may cause a different observed response than the aerosol of interest. (Cohen and Hering 1995)

## DIESEL AEROSOL SIZE INSTRUMENTATION

### Inertial Mobility Instruments

***Electrostatic Low-Pressure Impactor (ELPI):*** The ELPI is used extensively in Europe (Keskinen, et. al. 1992) to measure number distributions in near real-time. It can also be used as an inertial impactor to obtain mass size distributions by gravimetric analysis. The ELPI sizes particles by their aerodynamic diameter from about 30 nm to 10  $\mu\text{m}$  diameter. The ELPI is unlike a conventional inertial impactor in that it charges the particles prior to entering the impaction stage. Each impaction stage is connected to an electrometer that detects the current produced by the impacting particles. This current is converted into a particle concentration in the corresponding size range. This approach allows the instrument to make essentially real time size distribution measurements with a time resolution of about 2 s. The instrument is very sensitive and can measure ambient aerosol concentrations. It can also be used to measure size distributions during transient laboratory tests.

However, some uncertainties regarding calibration have been reported. In recent calibration experiments in United Kingdom comparing the ELPI with a scanning mobility particle sizer (SMPS – discussed below), the ELPI measured lower concentrations and larger particle diameters in the submicron diameter range (Dickens, et. al. 1997, Moon and Donald 1997). However, measurements made at the University of Birmingham and at the University of Minnesota show better agreement between the ELPI and SMPS (Keskinen, et. al. 1992) and more recently, measurements made by the Ford Motor Company also show good correlation between the ELPI and the SMPS (Maricq, 1998). Calibration difficulties in the upper end of the ELPI size range have also been reported by AEA laboratory in the United Kingdom. Another problem with the instrument is the rather high lower size cutoff of 30 nm. This is too large to detect the bulk of nanometer particles produced by new technology engines. The lower size limit is further compromised by the fact that the last few stages operate at rather low pressures (33, 17, and 8 kPa for channel midpoints of 115, 69, and 40 nm, respectively) that might result in evaporation of semi-volatile particles. Despite these limitations the ELPI is used for diesel particulate measurements because it is a versatile instrument, capable of spanning a wide range of particle size and concentration, in real-time, producing both number and volume size distributions. In particular, the ELPI is quite useful because it provides information over a wide size range, 30 nm to 10  $\mu\text{m}$ .

***Micro-Orifice Uniform Deposit Impactor (MOUDI):*** The MOUDI (Marple, et. al. 1991) is a 10 stage impactor with the four lower stages having micro-orifice impaction plates. The cut points at the nominal flow rate of 30 L/min are 10, 5.62, 3.16, 1.78, 1.0, 0.56, 0.316, 0.178, 0.10 and 0.056  $\mu\text{m}$  in aerodynamic diameter and uniform deposition is achieved by rotating the impaction plates using a motorized turner. The total pressure drop across the impactor is 0.3 atmospheres. Recent work at the University of Minnesota and elsewhere suggests that the combustion aerosol nuclei mode is composed primarily of volatile organic material, thus care must be take to avoid evaporative losses of volatile organics, particularly the condensed organic material. The MOUDI is a useful

instrument for determining the mass size distribution of diesel exhaust because it covers the entire size range. However, the MOUDI provides no size breakdown of aerosols < 50 nm nor does it provide data on the number distribution. Data are obtained after gravimetric analysis of the MOUDI impaction substrates so the MOUDI does not provide results in real-time. The diesel aerosol is normally diluted 10:1 or more before sampling to avoid overloading the impactor substrates from higher particulate concentrations. However, undiluted exhaust can be sampled directly if the sampling times are very short and care is taken not to overload the impaction substrates.

The MOUDI has been used extensively to determine the size distribution of diesel aerosols in underground mines (Cantrell and Rubow 1993). Underground mine size distributions are obtained under heavy engine load conditions from engine ranging in size from 100 to 700 hp. In mines, ambient temperatures span a broad range (45 to 100° F) as does humidity (<10 - 100 %). The dilution ratio typically ranges between 25-100:1, and is dependent upon location. Size distribution measurements obtained underground are discussed in the second supplemental report.

***Nano-Micro-Orifice Uniform Deposit Impactor (Nano-MOUDI):*** The nano-MOUDI (Marple, et. al. 1994) has 13 cut-sizes ranging from 0.010 to 18 µm and is designed to obtain four cut-sizes per decade. For cut-sizes above 50 µm, micro-orifice technology alone is sufficient to obtain the selected cut-points, but for cut-sizes below 50 µm micro-orifice nozzle technology is combined with low pressure operation. Gravimetric analysis determines the mass collected on aluminum foil substrates and a final stage filter. These substrates may be analyzed to determine the chemical composition of the mass collected on each stage.

## **Electric Mobility Instruments**

The heart of the electric mobility instruments is the electrical mobility analyzer. An aerosol is passed through a charger that imparts an electrical charge to the particles. The charged particles then pass through a variable electric field in the mobility analyzer which acts like a filter to precipitate out particles with a range of electric mobilities. The particles that are allowed to pass through the analyzer are then measured by an electrometer or a particle counter instrument. The strength of the electric field that causes particles with a specific electric mobility to precipitate on the analyzer surfaces can be varied to control the size of the particles that are allowed to pass through and be measured.

***Electrical Aerosol Analyzer (EAA):*** The EAA is the instrument that has been most commonly used in the U.S. (Liu *et al.*, Liu and Pui 1974a, Dolan *et al.*, Groblicki and Begeman, Fang and Kittelson, Baumgard and Kittelson, Baumgard and Johnson, Bagley *et al.*) for diesel aerosol size distribution measurements. FEV in Europe is also using an EAA for some of their sizing work

The EAA sizes particles by electrical mobility. Within the EAA, sampled particles are exposed to unipolar positive ions and become electrically charged. The charged particles

enter a mobility analyzer section of the EAA. The mobility analyzer functions as a low-pass filter, precipitating particles having high electrical mobility. Varying the applied voltage within the mobility analyzer allows the electrical mobility pass cut-point to be varied. The concentrations of particles that pass through the mobility analyzer are determined by measuring the current carried by the charged particles using an electrometer mounted downstream of the mobility analyzer section. Assuming that the electrical mobility of the sampled particles is a monotonic function of particle size, particle size cutpoints can be determined for each electrical mobility delineated by mobility analyzer voltage. A particle-size distribution can thus be generated by stepping the mobility analyzer through 11 voltages and measuring the current carried by the particles with an electrometer. A preclassification stage, such as an impactor or cyclone, must be used when sampling diesel exhaust in order to prevent the introduction of measurement errors from particles larger than 1.0  $\mu\text{m}$  (Yeh, 1993).

The measured particle-size range of the EAA is nominally from 3.2 nm to 1.0  $\mu\text{m}$ , but it is effectively 10 nm to 0.5  $\mu\text{m}$ . The sensitivity of the EAA drops off dramatically below 10 nm (Liu and Pui 1974a), due to diffusional particle losses and incomplete particle charging. The result is that the electrometer current for the first two voltage steps, which correspond to the 3.2 nm to 5.6 nm and 5.6 nm to 10 nm electrical mobility size ranges, respectively, contains only random signal noise unless extremely high particle number concentrations are present in those size intervals.

Approximately 2 to 3 minutes are required for the EAA to cycle through the 11 voltage steps. The time required for the voltage stepping poses a limit in using the EAA to measure the size distribution of a rapidly varying aerosol. The integral nature of EAA size measurement can also introduce errors if particle number concentrations change within the time interval of a single scan of EAA voltages. This has typically precluded the use of the EAA over transient speed and engine load test cycles such as the U.S. EPA Heavy-Duty Transient Test (40 CFR 86, Subpart N) without some means of collecting or integrating the particles over the cycle prior to analysis by the EAA. Hammerle, *et al.* (1994) and Dolan, *et al.* (1980) used bag samples to collect particles over transient test cycles for later size analysis by the EAA. Particle losses of up to 50% after 20 minutes were reported by Hammerle *et al.* (1994).

Diesel aerosol measurements with the EAA typically necessitate the use of relatively high dilution of diesel exhaust samples (300:1 to 1000:1) to prevent electrometer saturation. Although similar ranges of dilution have been reported for roadside dilution of vehicle exhaust, typical laboratory dilution tunnels operate with dilution ratios ranging from approximately 10:1 to 20:1, necessitating additional dilution of the exhaust sample prior to entering the EAA (Baumgard and Kittelson 1985), (Baumgard and Johnson 1992), (Bagley, *et al.* 1996).

The EAA requires a very steady concentration to produce accurate size distributions. If concentration shifts during a measurement, the instrument interprets that as an increased or reduced concentration in the size being measured at the time of the transient, even if the change is due to something occurring in another size range. The EAA has been

largely superseded by the SMPS (described below) because it is susceptible to measurement error of this type. However, it still provides useful measurements when properly applied. Furthermore, it provides a useful link to a body of work done over the past 20 years.

***Differential Mobility Particle Sizer (DMPS):*** The DMPS (Pui and Swift 1995) also sizes particles by electrical mobility using an electrical mobility analyzer. Sampled particles are exposed to a bipolar charger (rather than the unipolar charger of the EAA). The charged particles enter a mobility analyzer section of the DMPS and rather than acting as an integrating low-pass filter, only particles with a specific range of electric mobility are allowed to pass through the differential mobility analyzer and be measured. The applied voltage within the mobility analyzer is varied by discrete steps that allows the electrical mobility size interval to be varied. This technique is capable of higher resolution than the EAA, but measurement sensitivity using an electrometer is decreased. This is due to a smaller interval of particles leaving the analyzer, resulting in a smaller current flow to the electrometer. To avoid this reduction in sensitivity, the DMPS is typically operated with a high sensitivity condensation nuclei counter or condensation particle counter rather than an electrometer.

A preclassification stage, such as an impactor or cyclone, must be used when sampling diesel exhaust in order to prevent the introduction of measurement errors from particles larger than 1.0  $\mu\text{m}$  (Yeh, 1993).

***Scanning Mobility Particle Sizer (SMPS):*** The most common instrument in use for sizing engine exhaust in the submicrometer diameter region is the SMPS. This instrument is being used extensively in diesel engine test laboratories in the U.S. and Europe. It sizes particles by their electrical mobility equivalent diameter in the range from about 10 to 700 nm diameter. The lower limit can be extended to about 7 nm, but the uncertainty factor increases significantly. Although the SMPS can complete a scan of 100 size intervals in 30 s, results are distorted. Sampling times of 1-min per distribution still are subject to some distortion in shape. Size distributions obtained over 2 – 4 min are more typical of normal operating times and result in distributions with little distortion. The relatively slow scan time of the SMPS make it difficult to obtain size distributions under transient cycle conditions.

The SMPS consists of a pre-classification stage (such as an impactor or cyclone), used when sampling diesel exhaust in order to prevent the introduction of measurement errors from particles larger than 1.0  $\mu\text{m}$ , a DMA for particle classification, and a condensation nuclei counter (CNC) for particle counting.

The SMPS and similar instrumentation such as the scanning electrical mobility spectrometer (SEMS) (Wang and Flagan 1990), use continuous scans of mobility analyzer voltage rather than voltage stepping used by other instruments such as the EAA. The use of a low residence time, counting-type condensation nuclei counter (CNC) allows rapid, high resolution determination of submicrometer particle size distributions. The voltage of the mobility analyzer is ramped exponentially. The resultant monotonic

variation of the electric field strength in the mobility analyzer causes a monotonic variation in the electrical mobility of the particles entering the particle collection slot. After allowances are made for particle residence times, the entire size distribution can be derived from a single scan. Scans of 100 particle-size intervals can be completed in 30 s. Scanning the voltage both up and down to generate two size distribution measurements can be completed in 60 s. Because the voltage is scanned rather than stepped, the number of size intervals is primarily limited by particle residence time and the speed at which the particles can be counted by the CNC (Keady, et. al. 1983), Pollack counter (Hoppel 1978) or electrometer (Liu and Pui 1974) (Winklmayr, et. al. 1991). The size interval is therefore defined by number counts over a voltage interval within the mobility analyzer voltage scan, thus the operating characteristics of the SMPS are dependent upon the particle counter being used.

The SMPS can also be run in a single size mode as well as in a scanning mode. In the single size mode it measures a single particle size range continuously with a time resolution of a few seconds. In this mode it can be used for transient particle size measurements, one size range at a time (Greenwood, et. al. 1996). EMPA, Ford, and AEA are using this approach, although Ford has not applied the technique to diesel exhaust. The problem with this approach is that only one size range at a time is measured when a minimum of 3 to 5 size ranges are required to approximately describe the submicrometer size distribution. Therefore at least 3 to 5 transient tests are required to obtain one size distribution, which makes the approach time consuming and expensive. Alternatively, several instruments used in parallel, each measuring a different size, can be used, but this requires a large investment in instruments.

Size distribution measurements with the SMPS will be biased if particle concentration or size changes during the time interval of the scan. This has typically precluded the use of the SMPS over transient speed and engine load test cycles such as the U.S. EPA Heavy-Duty Transient Test without some means of providing a steady aerosol sample stream during the SMPS scan. A bag sampler that quickly fills and then allows an integrated, essentially constant, aerosol sample to be withdrawn during the SMPS scan could be used. Bag samples have been used to collect particles during transient test cycles and for on-highway studies (Chen, *et al.* 1984, Keady, *et al.*, 1983 and Kapadia, 1980) and if losses are characterized and analysis times minimized, this approach may essentially eliminate this problem.

[Authors' Note: Based on conversations with the manufacturer of the SMPS (Havlicek, 1998), versions 2.0 and 2.1 of the SMPS software contained an error that caused number counts of nanoparticles less than 10  $\mu\text{m}$  to be inaccurately high with CPC models 3022A and 3025A. The error has been corrected with the software version 2.3 and the manufacturer has distributed this version to owners of affected systems. Data generated by the SMPS is stored as raw data, allowing previous aerosol measurements performed with the inaccurate software version to be corrected with the updated software. Although the error with SMPS may have caused inaccurately high number concentrations of nanoparticles, results from studies of diesel exhaust with the correct SMPS software

(Abdul-Khalek, 1998) and other instruments (Bagley, 1996) still indicate the existence of a large nuclei mode of exhaust particles under certain conditions.]

Both the SMPS and EAA require a preclassifier to remove supermicron particles. The SMPS can operate at lower dilution ratios than EAA, such as the 10 - 30:1 ratios typically found in dilution tunnels. However, under these conditions the instrument fouling becomes a concern. The SMPS operates most effectively at dilution ratios of around 100:1 for typical diesel exhaust aerosols. Here is an example of how instruments used to obtain the aerosol size distribution affect the conditions under which the aerosol is collected. The size distributions obtained under 30:1 dilution may be quite different from those obtained from 1,000:1.

***Nanometer Differential Mobility Analyzer (nano-DMA):*** A new, nanometer differential mobility analyzer (*nano-DMA*) (Chen and Pui 1997) (Chen, et. al. 1996, 1998) (Hummes, et. al. 1996), optimized for nanometer size particles, scans the voltage over a narrower range of voltages and particle sizes. Using this narrow range voltage scanning, a CPC, and a flow geometry that minimizes residence time and diffusional losses, scans of particle size between 3 - 50 nm can be completed at high resolution in approximately 10 seconds. This particle size range roughly corresponds to the complete nuclei-mode of DPM. The nano-DMA is still in the experimental stage and has not been completely characterized and is not yet available commercially.

### **Photoemission Aerosol Instruments**

It has been recognized that the chemical composition of a diesel particle might influence its health impact when deposited in the human lung. Unburned hydrocarbons, such as polycyclic aromatic hydrocarbons (PAH's), from incomplete combustion are recognized as potent mutagens and carcinogens. Historically, analysis of the PAH content of diesel particle matter has been by particle collection on filters, extraction of the soluble organic fraction and PAH identification. Prolonged sampling times are required to obtain sufficient mass for analysis and this introduces potential errors through artifact formation. However, this method quantifies specific PAH compounds as illustrated by research at MTU (Bagley, *et al.*, 1996.)

Aerosol photoemission sensors have been developed for in-situ measurement of total particle bound PAH's and provide an alternative method for PAH quantification (Burtscher and Siegmann 1993) .

***Photoelectric Aerosol Sensor (PAS):*** Aerosol photoelectric sensors (Burtscher and Siegmann 1993, Burtscher, et. al. 1993, Hart, et. al. 1993) have been used to quantify the total PAH content associated with diesel particulate matter. In a typical configuration for a PAS, the aerosol passes through an electrical condenser that removes any charged particles or ions. The remaining neutral particles pass through a photoemission chamber where they are irradiated with an UV laser or flash lamp. Particles with photoelectric work functions below the photon energy of the UV source emit electrons and become positive ions (Weiss 1997). The photoemission chamber has a small electric field that

precipitates out the high mobility electrons and negative ions. The positive particles are collected on a filter and the resulting current measured by an electrometer. The intensity of the particle photoemission is linearly related to the amount of particle-bound PAH's and the particle surface area (Burtscher and Siegmann 1993).

PAS instruments measure the photoemission from particles which is related to particle surface and gives a surface area concentration. Results from the studies cited are all reported as photoemission intensity over time. More details on the relation of photoemission to PAH concentration, determined by chemical analysis are given elsewhere (Hart, *et al.*, 1993).

Only submicrometer particles may be irradiated and charged. Particles larger than 1  $\mu\text{m}$  are removed from the aerosol before it enters the photoemission chamber because they cannot be charged efficiently. Lower particle size limits and concentration limits for the PAS instruments have not been reported. This method does not quantify individual PAH compounds. The detection limit is about 1  $\text{ng}/\text{m}^3$  with a time resolution of about 1s (Burtscher and Siegmann 1993).

### **Mass Spectroscopy Instruments**

Mass spectroscopy has been used for decades to characterize the composition of aerosol particles. In principle, a particle is pulsed with a laser that desorbs/ionizes material that is measured by a mass spectrometer, indicating the chemical components of the particle (Gard, *et. al.* 1997, Noble and Prather 1996). Mass spectroscopy has an advantage over the photoemission method discussed above because it gives information for individual chemical components rather than total PAH content.

Two time-of-flight mass spectrometers are described below. While the instruments provide information on the chemical composition of particles, neither instrument is currently capable of providing size resolved chemical characterization in the lower end of the accumulation mode or the nuclei mode of diesel exhaust aerosols.

***Aerosol Time-of-Flight Mass Spectrometer (ATOFMS):*** The ATOFMS (Noble and Prather 1996 and 1998, Gard, *et. al.* 1997, Silva and Prather 1997) is unique in that it provides both real-time measurement of aerodynamic size and chemical composition of individual particles from a polydisperse aerosol. The instrument can analyze up to 600 particles per minute and can measure particles down to 0.3  $\mu\text{m}$  in diameter.

A particle enters the ATOFMS and is accelerated to terminal velocity by supersonic expansion of the carrier gas. Smaller particles reach a higher velocity than larger particles. The aerodynamic diameter is then determined by time-of-flight between two lasers before the particle enters the ionizing region of the mass spectrometer. Each particle is then pulsed by a laser. The resulting ions and molecules that are desorbed/ionized from the particle by the laser are measured by a mass spectrometer, indicating chemical components of the desorbed or ionized material. The spectrometer

includes two oppositely charged reflectrons that measure both positive and negative ions and provides good characterization of inorganics and light and heavy organics. Silva and Prather (1997) found the chemical composition of exhaust particles to be related to the size of the particle being measured and attribute this to the formation mechanism of the particle. Larger automobile exhaust particles were found to be inorganic while smaller particles were found to be mainly organic. However, all the particles measured by the ATOFMS in this study were in the accumulation mode and the nuclei mode particles were not characterized. They also reported finding platinum, cerium, and molybdenum in exhaust particles.

ATOFMS is an evolution from earlier mass spectral analysis of individual particles called *laser microprobe mass spectrometry (LMMS)*. LMMS uses mass spectroscopy of individual particles that are collected on a filter or impactor stage. Sampling times are on the order of 1 particle per minute, so it is not a real-time instrument. Samples are also subject to deterioration on the collection substrate before measurement (Noble and Prather 1996). Additional drawbacks of LMMS include that results are not reproducible and the theory of ion formation and behavior for the LMMS are not fully developed (Weiss 1997).

***On-Line Aerosol Mass Spectrometer:*** This instrument (Weiss, 1997) is similar in design to the ATOFMS and also provides both real-time particle sizing and composition information. The lower limit for aerodynamic sizing is around 0.3 to 0.4  $\mu\text{m}$  because particles smaller than this do not scatter sufficient light to be measure for aerodynamic sizing. Particles larger than the upper limit on particle size of about 50  $\mu\text{m}$  quickly clog the extremely small nozzles and orifices in the instrument. The on-line mass spectrometer can analyze up to 120-600 particles per minute.

The on-line mass spectrometer uses a single ion detector that characterizes inorganic and lighter organic material well but does not provide adequate resolution for more complex organics. It has been used to measure the concentration of cerium in diesel particulate matter that had been added to the fuel of a DI diesel engine. Metal based fuel additives such as cerium are added to diesel fuel so as to act as an oxidation catalyst to assist in the regeneration of diesel exhaust filters. Determination of the fate of these metals in diesel exhaust adds to the understanding of their impact on diesel emissions.

***Particle-Beam Mass Spectrometer (PBMS):*** (Ziemann, 1995, Kittelson, 1998) This method uses a mass spectrometer to determine the composition of the volatile organics and inorganics of particles that have been collected on an electrometer. An aerosol travels through a differential mobility analyzer that allows only particles of a selected size to pass through. A particle beam is then generated by sending the charged particles selected by the DMA through a sequence of openings that concentrate the particles into a narrowing beam. The particle beam is then directed at an electrometer that acts as a particle collector. When sufficient particle mass is collected, the electrometer is heated to about 200  $^{\circ}\text{C}$ , driving off the volatile organics and inorganics which are analyzed by a mass spectrometer.

This method allows the chemical characterization of the diesel particulate matter volatile material within a specific size. The collection of the particles allows sufficient mass for the mass spectrometry to be performed on nano-particles. There are currently no published results from this work.

### **Radioactive Tracer Instruments**

The attachment of a radioactive tracer to particles allows the measurement of particle radioactivity to be related to particle surface area.

***Epiphaniometer:*** The epiphaniometer (Gaggeler, et. al. 1989a, Gaggeler, et al 1989b) is an instrument developed at the Paul Scherrer Institute in Switzerland that measures the surface concentration of aerosol particles in both the nuclei and accumulation mode size ranges. The epiphaniometer is most sensitive to particles in the accumulation mode, but Gaggeler, et. al. 1989a reported successfully measuring silver particles between 20 and 90 nm that were agglomerates formed from smaller primary silver particles. The maximum concentrations the epiphaniometer can handle is not reported, but it is capable of measuring low atmospheric particle concentrations found in remote locations.

In an epiphaniometer, aerosol is passed through a charging chamber where lead isotopes created from a decaying actinium source are attached to the particle surfaces. The particles are transported through a capillary to a collecting filter. The epiphaniometer uses a surface barrier detector to measure the level of radioactivity of the particles collected on the filter. The amount of radioactivity is proportional to the particle's Fuchs surface area and follows Fuchs theory of attachment of radioactive isotopes.

Because of the short half-life of the lead isotopes, the filter does not become saturated and essentially real-time radioactivity measurements can be made. Although not clearly stated in the studies, the surface barrier detector measures radioactivity and must be related to the size of particles being sampled to get a measure of surface area of the particles. A pre-classifier, such as a DMA, may be used before the epiphaniometer to allow a determined range of particles to enter the instrument.

### **Other Sizing Instruments**

Other instruments have been applied to exhaust particle measurements by European labs, especially by AEA where a systematic study of a variety of instruments has been done (Dickens, et. al. 1997). These instruments include a quartz crystal substrate impactor, the Stober centrifuge, and Anderson impactors. Of these, only the quartz crystal impactor gives near real time measurements. The centrifuge and the Anderson impactor both require substrate removal and weighing to determine size distributions. The quartz crystal impactor grossly underestimates the concentrations of diesel particles throughout its operating range. Apparently diesel particles do not couple well with the vibrating surfaces of the quartz substrates. This causes the instrument to underestimate deposited mass. The U. S. Bureau of Mines encountered exactly the same problem many years ago (Welker, et al, 1982).

Ricardo in England has tried to use an aerodynamic particle sizer (APS) for exhaust particle measurements. This instrument sizes particles by aerodynamic diameter in the size range from about 0.5 to 10  $\mu\text{m}$  diameter. The problem with using an instrument of this type for engine size measurements is that it misses the size range where most of the mass, and even more so, number of diesel exhaust particles are found. Furthermore, if there is a large concentration of particles just below the size detection limit of the instrument it can result in a shadow mode just above the lower detection limit of the instrument that is not really there (Baron, Mazumder and Cheng, 1993, Heitbrink, *et al.* 1991, Baron, 1986).

**Diffusion Batteries:** Diffusion batteries were developed to determine particle diffusion coefficients (Cheng 1993), and have been used for size characterization of 1 nm to 100 nm aerosols for more than 50 years (DeMarcus and Thomas 1952, Thomas 1955) by deriving particle size from diffusion characteristics. Diffusion batteries separate particles by their diffusion mobilities. Mathematical expressions have been derived which describe particle concentration profiles for various flow profiles within, geometries of, screens, tubes, and beads that are commonly used for diffusion batteries (Cheng 1993, Cheng *et al.* 1985, Cheng and Yeh 1984). A number of numerical inversion techniques have been applied to the penetration versus particle size of polydisperse aerosols within diffusion batteries of varying geometries (Cheng and Yeh 1984, Kapadia 1980, Maher and Laird 1985, Twomey 1975). Diffusion batteries are typically used with a switching valve to vary the effective diffusional path length, and a CNC or Pollack counter for measuring particle number concentrations (Sinclair 1986, Sinclair *et al.* 1979)

Screen type diffusion batteries are commercially available with switching valves for varying the effect diffusional length. By switching the valve and counting the particles using a CNC, a particle size distribution can be obtained between approximately 5 nm and 0.5  $\mu\text{m}$ . Switching diffusion batteries have been used to determine particle size vs. number concentration for diesel PM (Dolan *et al.* 1980, Baumgard and Kittelson, Clary), and have typically shown good agreement with electrical mobility sizing methods and inertial impaction sizing methods over the size ranges that overlap with these types of instruments. The time necessary for switching the effective length of the diffusion battery and for CNC measurement limits switching diffusion battery/CNC combinations to the measurement of particle size over relatively steady-state conditions.

**Parallel Flow Diffusion Battery (PFDB):** A PFDB (Cheng and Yeh 1984, Cheng, *et al.* 1984, Degobert 1988) has been used to measure particle-size in diesel exhaust in the 3 nm to 0.5  $\mu\text{m}$  size range over the light-duty, FTP test cycle using a standard, single dilution, EPA-style CVS dilution tunnel at dilution ratios of approximately 8:1 to 10:1 (Cheng and Yeh 1984). The PFDB uses seven parallel flow paths, each with a varying number of stacked, stainless steel mesh screens followed by a 25 mm Zefluor<sup>TM</sup> filter. Critical flow orifices are used to control the sample flow rate through each set of stacked screens and filters. The PFDB was used with a low pressure cascade impactor to allow classification of larger particles and to provide a necessary 0.7  $\mu\text{m}$  preclassification cutpoint (Cheng and Yeh 1984). A similar device was developed (Boulad and Diouri

1988) and used for DPM size characterization (Degobert 1988) with an 8-stage cascade impactor from 0.35  $\mu\text{m}$  to 7.5  $\mu\text{m}$ , and diffusion battery with 5 parallel bead-beds covering 8 nm to 0.35  $\mu\text{m}$ . Advantages of this type of diffusion battery measurement procedure include; direct filter mass measurement procedures, ability to integrate particle mass measurements for each size range to be over transient engine operating conditions, and similarity to actual diffusion-deposition mechanism for fine particles in lung tissue. Disadvantages include; potential difficulty in resolving the (typically) very small mass concentration of ultra-fine nuclei-mode particles, and errors associated with the data inversion techniques. It might be possible to combine the parallel diffusion battery approach with particle detection using CNCs. This would give near real time response, but would require multiple CNCs.

### **GM AND FORD LAB VISITS**

The investigators recognized that the dilution, sampling procedures and aerosol instruments used in diesel laboratories determine the aerosol size distributions. With this in mind, Profs. Kittelson and Johnson visited the engine laboratories of GM and Ford in Detroit in September, 1997 to gather information on how these organizations were sizing diesel aerosol.

The Ford laboratory has dual stainless steel CVS type tunnels, one for diesel, one for gasoline. They are very concerned about condensation in the tunnels. The inlet air is dry and heated to prevent condensation during cold starts with gasoline engines. Particle sizing instruments include a SMPS in conjunction with a 3010 CNC and an ELPI. No secondary dilution is used for gasoline engines and Ford has not tested diesels yet. Since the SMPS has a 30 s scan time, but can obtain a sample for a single channel in 1 s transient size distributions are measured by running on 4 consecutive days, each day with the SMPS set to a different size channel. This is time consuming and expensive. Recent work reported by Maricq (1998) showed good agreement between the SMPS and ELPI.

Most of the engine exhaust particle measurement work at GMR in recent years has been associated with Coordinating Research Council sponsored field studies, which have determined particle mass emissions from in-use vehicles. GMR is refurbishing their lab for particle size measurements. They have a CVS type dilution tunnel operating with filtered laboratory air. Particle sizing instruments consisted of a MOUDI and an EAA.

As a result of these lab visits, a questionnaire was prepared and sent to the major engine labs in the U.S. and Europe to obtain current data. The responses from the questionnaire are given in Appendix A.

### **REFERENCES**

Baron, P. A. 1986. Calibration and Use of the Aerodynamic Particle Sizer (APS 3300). *Aerosol Sci. Technol.* 5(1): 55-67.

Baron, P. and K. Willeke. 1993. Chapter 2 Aerosol Fundamentals. In *Aerosol Measurement Principles Techniques and Applications*, K. Willeke and P. Baron editors, Van Nostrand Reinhold, NY, NY., p. 8-22.

Baron, P. A., M. K. Mazumder and Y. S. Cheng. 1993. Chapter 17 Direct Reading Techniques Using Optical Particle Detection. In *Aerosol Measurement Principles Techniques and Applications*, K. Willeke and P. Baron editors, Van Nostrand Reinhold, NY, NY., p. 381-409.

Baumgard, K.J. and J. H. Johnson. 1992. The Effect of Low Sulfur Fuel and a Ceramic Particle Filter on Diesel Exhaust Particle Size Distributions. Technical Paper Series, No. 920566, SAE: Warrendale, PA.

Baumgard, K.J. and D. B. Kittelson. 1985. The Influence of a Ceramic Particle Trap on the Size Distribution of Diesel Particles. Technical Paper Series, No. 850009, SAE: Warrendale, PA.

Bagley, S. T., K. J. Baumgard, L. D. Gratz, J. H. Johnson, and D. G. Leddy. 1996. Characterization of Fuel and Aftertreatment Device Effects on Diesel Emissions. Health Effects Institute Research Report Number 76, 88 pp.

Boulaud, D. and M. Diouri. 1988. A New Inertial and Diffusional Device (SDI 2000). *J. Aerosol Sci.*, Vol. 19(7), pp. 927-930.

Burtscher, H., D. Matter, and D. Steiner. 1993. Characterization of Soot Particles by In-Situ Measurement with Different Aerosol Analysis Tools. *J. Aerosol Sci.* , Vol. 23, pp. 65-67.

Burtscher, H. and H. C. Siegmann. 1993. Photoemission for In Situ Analysis of Particulate Combustion Emissions. *Water, Air, and Soil Pol.*, Vol. 68: 125-136.

Cantrell, B. K. and K. L. Rubow. 1993. Measurement of Diesel Exhaust Aerosol in Underground Coal Mines. Bureau of Mines IC 9324, p. 11-17.

Chen, D. R. and D.Y.H Pui. 1997. A Novel Unipolar Aerosol Charger. Annual meeting of the AAAR, Denver, CO, October 13-17.

Chen, D. R., D.Y.H. Pui, D. Hummes, H. Fissan, F. R. Quant and G. J. Sem. 1996. Nanometer Differential Mobility Analyzer (Nano-DMA): Design and Numerical Modeling. *J. Aerosol Sci.* Vol. 27 Supplement 1 pp. S137-S138.

Chen, D. R., D.Y.H. Pui, D. Hummes, H. Fissan, F. R. Quant and G. J. Sem. 1998. Design and Evaluation of a Nanometer Aerosol Differential Mobility Analyzer (Nano-DMA). *J. Aerosol Sci.* Vol. 29, No. 5/6, pp. 497-509.

Cheng, Y.S. 1993. Condensation Detection and Diffusion Size Separation Techniques. In Aerosol Measurement, K. Willeke and P.A. Baron, eds. Van Nostrand Reinhold: New York, NY, pp. 427-451.

Cheng, Y.S. and C. H. Yeh. 1984. Analysis of Screen Diffusion Battery Data. Am. Ind. Hyg. Assoc. J., 45(8):556-561.

Cheng, Y.S., H. C. Yeh, and K. J. Brinsko. 1985. Use of Wire Screens as a Fan Model Filter. Aerosol Sci. Tech., 4:165-174.

Cheng, Y.S., C.H. Yeh, J.L. Mauderly and B. V. Mokler. 1984. Characterization of Diesel Exhaust in a Chronic Inhalation Study. Am. Ind. Hyg. Assoc. J., 45:547-555.

Clerc, J.C., and J. H. Johnson. 1982. A Computer Heat Transfer and Hydrocarbon Adsorption Model for Predicting Diesel Particulate Emissions in Dilution Tunnels. SAE Technical Paper Series, No. 821218.

Cohen, B. S. and S.V. Hering (eds.). 1995. Air Sampling Instruments for Evaluation of Atmospheric Contaminants. American Conference of Governmental Hygienists, Inc., 651 pp.

Degobert, P. Emissions Diesel, Aspects Physico-Chimiques. 1988. 5<sup>ème</sup> Symp. Sur la Recherche en Matière de Pollution Atmosphérique, Strasbourg, March 22-25, 13 p, (in French).

DeMarcus, W. and J. W. Thomas. Theory of a Diffusion Battery. 1952. Report No. ORNL-1413, Oak Ridge National Laboratories: Oak Ridge, TN.

Dickens, C. J., M. H. E. Ball, D. Booker, J. R. Donald, A. M. Tope and M. Hughes. 1997. Evaluation of Instruments for Vehicle Emission Particle Sizing. AEA Technology Report AEAT-1180, 69 pp. (restricted distribution).

Dolan, D.F., D.B. Kittelson, and D.Y.H. Pui. 1980. Diesel Exhaust Particle Size Distribution Measurement Techniques. SAE Technical Paper Series, No. 800187.

Dolan, D.F., D. B. Kittelson, and K. T. Whitby. 1975. Measurement of Diesel Exhaust Particle Size Distributions. Paper No. 75-WA/APC-5, American Society of Mechanical Engineers: New York, NY.

Fang, C.P., and D. B. Kittelson. 1984. The Influence of a Fibrous Diesel Particulate Trap on the Size Distribution of Emitted Particles. SAE Technical Paper Series, No. 840362.

Gaggeler, H.W., U. Baltensperger, M. Emmenegger, D. T. Jost, A. Schmidt-Ott, P. Haller, and M. Hofmann. 1989a. The Epiphaniometer, A New Device for Continuous Aerosol Monitoring. J. Aerosol Sci., Vol. 20, No. 5, pp. 557-564.

- Gaggeler, H.W., U. Baltensperger, D.T. Jost, M. Emmenegger, and W. Nageli. 1989b. Applications of the Epiphaniometer to Environmental Aerosol Studies. *J. Aerosol Sci.*, Vol. 20, No. 8, pp. 1225-1228.
- Gard, E., J.E. Mayer, B. D. Morrical, T. Dienes, D. P. Ferenson, and K. A. Prather. 1997. Real-time Analysis of Individual Atmospheric Aerosol Particles: Design and Performance of a Portable ATOFMS. *Anal. Chem.*, Vol. 69, No. 20, pp. 4083-4091.
- Greenwood, S.J., J. E. Coxon, T. Biddulph and J. Bennett. 1996. An Investigation to Determine the Exhaust Particulate Size Distributions for Diesel, Petrol, and Compressed Natural Gas Fuelled Vehicles. SAE Technical Paper Series, No. 961085.
- Groblicki, P.J. and C. R. Begeman. 1979. Particle Size Variation in Diesel Car Exhaust. SAE Technical Paper Series, No. 790421.
- Hammerle, R. H., D. A. Ketcher, R. W. Horrocks, G. Lepperhoff, G. Hüthwohl, and B. Lüers. 1994. Emissions from Current Diesel Vehicles. SAE Technical Paper Series, No. 942043.
- Hart, K.M., St. R. McDow, W. Giger, D. Steiner, and H. Burtscher. 1993. The Correlation Between In-Situ, Real-Time Aerosol Photoemission Intensity and Particulate Polycyclic Aromatic Hydrocarbon Concentration in Combustion Aerosols. *Water, Air, and Soil Pollution*, Vol. 68, pp. 75-90.
- Havlicek, M. 1998. Personal Communication with M. Arnold. TSI, Inc.
- Heitbrink, W. A., P. A. Baron, and K. Willeke. 1991. Coincidence in Time-of-flight Spectrometers: Phantom Particle Creation. *Aerosol Sci. Technol.* 14:112-126.
- Hering, S.V. 1995. Impactors, Cyclones, and Other Inertial and Gravitational Collectors. Ch. 14 in *Air Sampling Instruments for Evaluation of Atmospheric Contaminants*. Ed. by B. S. Cohen, and S.V. Hering. ACGIH, Cincinnati, OH, 8<sup>th</sup> Edition, pp. 279 – 305.
- Hoppel, W. A. 1978. Determination of the Aerosol Size Distribution from the Mobility Distribution of the Charged Fraction of Aerosols. *J. Aerosol Sci.*, 9:41-54.
- Hummes, D., S. Neumann, H. Fissan, D. R. Chen, D. Y. H. Pui, F. R. Quant and G. J. Sem. 1996. Nanometer Differential Mobility Analyzer (Nano-DMA): Experimental Evaluation and Performance Verification. *J. Aerosol Sci.* Vol. 27 Supplement 1 pp. S136-S137.
- Keady, P.B., F. R. Quant, G. J. Sem. 1983. *TSI Quarterly* (9):3.
- Kapadia, A. 1980. Data Reduction Techniques for Aerosol Size Distribution Measurement Instruments. Ph. D. Dissertation, University of Minnesota.

- Keskinen, J., K. Pietarinen, M. Lehtimäki. 1992. Electrical Low Pressure Impactor. *J. Aerosol Sci.* 23(4):353-360.
- Kittelson, D. B. 1998., Personal communication with P. Ziemann University of California – Riverside.
- Kittelson, D. B., W. F. Watts, and M. J. Arnold. 1998a. Diesel Exhaust Particle Measurement Instruments - Supplemental Report No. 2 to EPA Grant Aerosol Dynamics, Laboratory and On-Road Studies.
- Kittelson, D. B., M. J. Arnold, and W. F. Watts. 1998b. Review Of Diesel Particulate Matter Sampling Methods. Final Report EPA Grant.
- Liu, B.Y.H and D. Y. H. Pui. 1974a. On the Performance of the Electrical Aerosol Analyzer. *J. of Aerosol Sci.*, 6:249-264.
- Liu, B.Y.H. and D. Y. H. Pui. 1974b. A Submicron Aerosol Standard and the Primary, Absolute Calibration of the Condensation Nucleus Counter. *J. of Colloid Interface Sci.*, 47:155.
- Liu, B.Y.H., K. T. Whitby and D. Y. H. Pui. 1974. A Portable Electrical Analyzer for Size Distribution Measurement of Submicron Aerosols. *JAPCA.*, 24(11):1067-1072.
- Maher, E.F. and N. M. Laird. 1985. EM Algorithm Reconstruction of Particle Size Distributions from Diffusion Battery Data. *J. Aerosol Sci.*, 16:557-570.
- Maricq, M. 1998. Vehicle Particulate Emissions: A Comparison of ELPI, SMPS and Mass Measurement. Included in Presentation Handouts (Presented at International Seminar on Particle Size Distribution Measurement From Combustion Engines, Espoo, Finland, May 18-19, 1998.)
- Marple, V. A, K. L. Rubow and S. M. Behm. 1991. A Microorifice Uniform Deposit Impactor (MOUDI): Description, Calibration, and Use. *J. Aerosol Sci. Technol.*, 14:434-446.
- Marple, V. A., K. L. Rubow, and B. A. Olson. 1994. Low Pressure Stages for the Micro-orifice Uniform Deposit Impactor (MOUDI). Fourth International Aerosol Conference Abstracts, ed. R.C. Flagan, Vol. 2, 6 pp.
- Moon, D. P. and J. R. Donald. 1997. UK Research Program on the Characterization of Vehicle Particulate Emissions. (Contract Report for the Department of the Environment, Transport and the Regions and the Society of Motor Manufacturers and Traders by ETSU – a group within AEA). ETSU-R98, 90 pp.

- Noble, C. A. and K. A. Prather. 1996. Real-time Measurement of Correlated Size and Composition Profiles of Individual Atmospheric Aerosol Particles. *Environ. Sci. Technol.*, Vol. 30, pp. 2667-2680.
- Noble, C. A. and K. A. Prather. 1998. Aerosol Time-of-Flight Mass Spectrometry: A New Method for Performing Real-time Characterization of Aerosol Particles. *Appl. Occup. Environ. Hyg.* 13(6):439-443.
- Pui, D.Y.H. and D.L. Swift. 1995. Direct-Reading Instruments for Airborne Particles. Ch. 16 in *Air Sampling Instruments for Evaluation of Atmospheric Contaminants*. Ed. by B. S. Cohen, and S. V. Hering. ACGIH, Cincinnati, OH, 8<sup>th</sup> Edition, pp. 337 – 368.
- Reichel, S., F. F. Pischinger and G. Lepperhoff. 1983. Influence on Particles in Diluted Diesel Engine Exhaust Gas. SAE Technical Paper Series, No. 831333.
- Silva, P.J. and K.A. Prather. 1997. On-line Characterization of Individual Particles from Automobile Emissions. *Environ. Sci. Tech.*, Vol. 31, pp. 3074-3080.
- Sinclair, D. 1986. Measurement of Nanometer Aerosols. *Aerosol Sci. Technol.*, 5:187-204.
- Sinclair, D., R. J. Countess, B. Y. H. Liu, and D. Y. H. Pui. 1979. Automatic Analysis of Submicron Aerosols in Aerosol Measurement (D. A. Lundgren *et al.*, eds.), University of Florida Presses, Gainesville, Florida, pp. 544-563.
- Thomas, J.W. 1955. The Diffusion Battery Method for Aerosol Particle Size Determination. *J. Colloid Sci.*, 10:246-255.
- Twomey, S. 1975. Comparison of Constrained Linear Inversion and an Alternative Nonlinear Algorithm Applied to the Indirect Estimation of Particle Size Distribution. *J. Comp. Phys.*, 18:188-200.
- Wang, S.H. and R. C. Flagan. 1990. Scanning Electrical Mobility Spectrometer. *Aerosol Sci. Tech.*, 13:230-240.
- Welker, R. W., *et al.* 1982. Mine Particulate Size Characterization. U. S. Bureau of Mines Open File Report, Prepared by IIT Research report number C06572-Final, p. 96.
- Weiss, M. 1997. An On-line Mass Spectrometer for Aerosols: Development, Characterization and Applications. Ph. D. Dissertation, Delft University of Technology, Netherlands.
- Winklmayr, W., G. P. Reischl, A. O. Lindner and A. Berner. 1991. A New Electromobility Spectrometer for the Measurement of Aerosol Size Distributions in the Size Range From 1 to 1000 nm. *J. Aerosol Sci.*, 22(3):289-296.

Yeh, H. 1993. Chapter 18 Electrical Techniques. In *Aerosol Measurement Principles Techniques and Applications*, K. Willeke and P. Baron editors, Van Nostrand Reinhold, NY, NY., p. 410-426.

Ziemann, P., *et al.* 1995. Measurement of Particle Induced Nucleation and Growth in Low Pressure Chemical Vapor Deposition Systems with the Particle Beam Mass Spectrometer. *J. Aerosol Sci.* Vol. 26, Suppl. 1, p. S633-S634

## Appendix 1 - Lab Survey Summary

### 1. Fuel Properties

Laboratory	Response
AEA	No formal specs - fuel as requested by clients
Biel	Swiss commercial diesel fuel: norm SN EN 590 KO ESSO Density: 0.815-0.845 kg/l at 15 C Lower heating value: $\geq 42.5$ MJ/kg Sulfur: $\leq 0.05$ %
EMPA	CEC RF 03 -A-84 Density: 0.841 kg/dm <sup>3</sup> C:H: 6.5242:1 Sulfur: < 0.05 %  OKK (Commercial) Density: 0.832 kg/dm <sup>3</sup> C:H: 6.0049:1 Sulfur: < 0.04 %
MTU	Sulfur: 310 ppm API gravity: 35.4 Cetane Index: 47.8
FEV	Different fuels are used (depending on the purposes of the project), e.g.: commercial EU fuels, Swedish or Japanese diesel or gasoline fuels, diesel with different contents of sulfur (artificial).
Ford	*
EPCD	Fuel: D-2 Specific gravity: 0.84799 g/ml C:H ratio: 0.5489 Lower heating value: 18443 Btu/lb Sulfur: 0.0359 wt %
UMN	Fuel: D-2 Specific gravity: 0.84 87 % C, 13 % H (by mass), C/H = 6.69 (mass ratio) Lower heating value: 43,000 Kj/Kg Sulfur: 0.04 wt %

\*Indicates no response

## 2. Lubricating Oil

Laboratory	Response
AEA	Mineral based such as Gastrol GTX
Biel	Synthetic such as DEA DES-6089
EMPA	No special oil, not changed for tests
MTU	Cummins premium Blue 2000, SAE 15W-40
FEV	Different lube oils are used (depending on the purposes of the project); most commonly mineral based oils.
Ford	Not recorded
EPCD	SAE 15 W- 40
UMN	SAE 15 W-40

## 3. Residence time from tailpipe to primary dilution tunnel

Laboratory	Response
AEA	All testing is carried out on a chassis dynamometer. The residence time depends upon the vehicle's exhaust output which varies throughout a drive cycle. Measurements of air velocities into the cleaner of a 2.7 l non-turbocharged diesel engine powered vehicle, driven at 50 and 110 kph, and subsequent calculation of exhaust outlet volumes, indicate residence times of 0.5 s and 0.3 s respectively.
Biel	From turbocharger to dilution tunnel inlet: 0.07 s (2,000 rpm at 100% load = max power)
EMPA	Strong dependence on vehicle and load, strong variation for time with transient tests typical range 0.4 - 4s
MTU	1.9 - 2.5 s from turbocharger to DT inlet depending upon load; time to particle filters 1.34 s; time to particle size instruments 1.8 s
FEV	The residence time depends on the exhaust volume flow and it is between 0.62 sec and 1.88 sec (@ 1,000 to 3,000 rpm)
Ford	Heated corrugated stainless steel hose, heated to 220 F: Time from tailpipe to tunnel inlet: Lower tunnel: 19 ft long, 3.6 in ID => 1.34 ft <sup>3</sup> => 8 s at 10 scfm, 0.8 s at 100 scfm Upper tunnel: 25 ft long, 4.1 in ID => 2.29 ft <sup>3</sup> => 13.8 s at 10 scfm, 1.4 s at 100scfm 1.5 s to particle filters; 3.0 s to particle size measurement instruments
EPCD	1.54 s from exhaust transfer tube to DT
UMN	*New Tunnel: 0.0 15 s from engine exhaust to primary at a dilution ratio of about 18 Old Tunnel: 0.4 sec from engine exhaust to primary at a dilution ratio of about 18 * The new tunnel is currently under construction.

#### 4. Type of transfer line/exhaust pipe

Laboratory	Response
AEA	Separate exhaust transfer lines are used for diesel and gasoline. They are made of 70 mm diameter smooth bore stainless steel tube and are insulated. The minimum length possible of flexible corrugated stainless steel tube, also insulated, is used to make the final connection to the vehicles' exhaust pipe. Path length varies between 2.5 and 5 m depending upon the vehicle being tested.
Biel	Exhaust pipe: Straight steel, 90° arch 3-4 times, unheated, 3.7 m without particle filter ~ 4.5 m with particle filter, ID 100 mm Dilution tunnel pipe: straight steel, heated 180° C, 35 mm long, ID 5 mm
EMPA	Engine dyno: about 6m stainless steel insulated, Ø 125 mm Chassis dyno: 0-2m stainless steel insulated, Ø 60mm; 2-5m stainless steel heated and insulated, Ø 100mm; 5-5.5m stainless steel, Ø 100mm => total length: 5.5 m different exhaust pipes for diesel and gasoline
MTU	Exhaust pipe diameter: 5 in; length from turbocharger to transfer line, 11.5 ft; transfer line diameter: 3 in and 65 in long
FEV	The transfer line consists of a unheated, insulated, straight stainless steel pipe with a length of 4 m and an unheated, insulated hose with a length of 3.6 m.
Ford	Heated corrugated stainless steel hose, heated to 220 F Lower tunnel: 19 ft long, 3.6 in ID Upper tunnel: 25 ft long, 4.1 in ID
EPCD	length of exhaust tube: 26 feet length of insulation: 24 feet exhaust tube diameter: 6 inches primary tunnel diameter: 18 inches No flexible tubing is used if it can be avoided.
UMN	New Tunnel: Straight insulated tube, 4.5 mm ID, 300 mm in length Old tunnel: Straight insulated and heated (300°C) Stainless Steel, 4.5 mm ID, 1250 mm in length

#### 5. Exhaust temperature at the tailpipe or at the exhaust transfer line.

Laboratory	Response
AEA	Most tests are carried out on a chassis dyno following the standard ECE and EUDC drive cycles. Hence the temperatures of the exhaust varies throughout the cycle. However, for a 2.7 l non-turbocharged diesel powered vehicle the temps are ~14 °C at 50 kph and ~240°C at 110 kph.
Biel	Before turbocharger 600 °C max with full load, after turbo at transfer line 520°C max
EMPA	Chassis: typical range: 90 - 300 °C Engine dyno: typical range: 200 - 400 °C
MTU	Tailpipe: 270-400 °C, Tunnel inlet: 190 - 300 °C, Dilution air: 20 - 27 °C
FEV	The temperature is different and depends on test condition (type of the car, velocity, etc.)
Ford	Dilution air: 28 °C
EPCD	425 - 482°C (max.) exhaust temperature at the transfer tube
UMN	New Tunnel: Exhaust Temperature 175- 550 °C Old Tunnel: 300 °C

## 6. Temperature of exhaust at the inlet of the primary dilution tunnel

Laboratory	Response
AEA	Not measured
Biel	450 °C max
EMPA	Not measured
MTU	Tailpipe: 270-400 °C, Tunnel inlet: 190 - 300 °C, Dilution air: 20 - 27 °C
FEV	The temperature is different and depend on test condition (vehicle/engine type, velocity). For diesel vehicle max. 300 °C.
Ford	Not measured
EPCD	Not measured
UMN	New Tunnel: Exhaust Temperature Old Tunnel: 300 °C

## 7. Physical characteristics of the primary dilution tunnel

Laboratory	Response
AEA	The primary dilution tunnel is constructed of smooth bore stainless steel, 22 cm ID by 2.5 m long. The tunnel is earthed and the particulate sizing sampling point is ~2.2 m downstream of the tunnel entrance.
Biel	AVL Smart Sampler 2, Model 472, partial flow system; Tunnel diameter: 70 mm, length 700 mm
EMPA	Chassis: <ul style="list-style-type: none"> <li>• construction material: stainless steel,</li> <li>• electrical grounding: yes</li> <li>• inner diameter: ≈ 320 mm</li> <li>• length: ≈ 4000 mm</li> <li>• sampling about 4 m downstream of the mixing point</li> </ul> Engine Dyno: <ul style="list-style-type: none"> <li>• construction material: stainless steel,</li> <li>• electrical grounding: yes</li> <li>• inner diameter: ≈ 450 mm</li> <li>• length: ≈ 5000 mm</li> <li>• sampling about 4.6 m downstream of the mixing point</li> </ul>
MTU	16 gauge 304 stainless steel, diameter = 0.305 m, distance from exhaust inlet to sampling location is 4.57 m
FEV	The construction material consists of straight stainless steel and an electrical grounding. The inner diameter of the dilution tunnel is 320 mm, and its length is 4 m.
Ford	*
EPCD	construction material: stainless steel electrical grounding: yes inner diameter: 18 inches length: 13.8 feet
UMN	New tunnel: Stainless steel probe with a short transfer line connected t o a critical orifice upstream of an ejector pump model Air Vac TD110SS Old tunnel: same as above with a longer heated and insulated transfer line

**8. Describe the secondary dilution system**

Laboratory	Response
AEA	*
Biel	*
EMPA	Chassis: not used; Engine Dyno stainless steel, 125 mm diameter, 500 mm length, no ejector diluter
MTU	Filtered compressed air, 30 psi, added in 2 steps, flow controlled by orifices
FEV	Depending on the kind of investigation secondary ejector dilution is used. The distance from the dilution tunnel to the measurement instrumentation including the ejector diluter is 2 m
Ford	*
EPCD	<ul style="list-style-type: none"> <li>- dilution tunnel?       yes</li> <li>- construction material: stainless steel</li> <li>- inner diameter:       4.25 inches</li> <li>- length                   32 inches</li> <li>- ejector diluter?       used only for particle sizing experiments</li> </ul>
UMN	<p>New Tunnel: capable of providing a range of residence times from 0.1-8 sec at the primary dilution ratio. The rest of the system is similar to the primary tunnel</p> <p>Old tunnel: Similar to the primary with a fixed residence time between primary and secondary of about 0.4 sec</p>

**9. What are the ranges of primary and secondary dilution? What are the dilution ratios for each bag sample of light-duty FTP tests?**

Laboratory	Response
AEA	The only data are from a 5.2 l CNG fueled vehicle. The primary dilution ratios were 14.1, 19.4., 14.7 to 1 for bags 1-3, respectively. No secondary dilution ratios available.
Biel	For Gravimetric Filters: total flow = 2 g/s, dilution flow = 1.6 g/s, ratio = 1:5 For Particle Size Measurements: total flow = 2 g/s, ratio = different
EMPA	<p>Chassis:</p> <ul style="list-style-type: none"> <li>• primary dilution ratios: 4-50</li> <li>• typical value: 20</li> </ul> <p>Engine Dyno:</p> <ul style="list-style-type: none"> <li>• primary dilution ratios: 2 - 30</li> <li>• secondary dilution ratios: 1.2 - 2</li> </ul>
MTU	Primary 8-12, Secondary 70-100
FEV	The dilution ratios are > 1:8
Ford	Bag 1 13.0, Bag 2 20.1, Bag 3 14.8 for typical FTP test with a 3.8 l engine
EPCD	<p>primary tunnel:           10:1 to 15:</p> <p>secondary tunnel:       approx. 2.5:1</p>
UMN	Old and New tunnel: Primary: 10-50; Secondary: 10-100

**10. What is the temperature and relative humidity of the dilution air entering the primary and secondary dilution tunnels? Are these controlled in any manner?**

Laboratory	Response
AEA	Not controlled, routine values are 20 - 25 °C and 35 - 55 % RH
Biel	Not controlled
EMPA	Primary dilution: <ul style="list-style-type: none"> <li>• 298.15K ± 5K, not controlled, but no problems</li> <li>• 50% ± 10%, controlled</li> </ul>
MTU	Primary dilution: <ul style="list-style-type: none"> <li>• 20-27 °C</li> <li>• Ambient relative humidity and steam to achieve 40 % at sampling location</li> <li>• Hydrocarbons removed by scrubber</li> <li>• Temperature of mixed air 46 ± 1 °C, 40 % RH</li> </ul> Secondary dilution: <ul style="list-style-type: none"> <li>• Compressed air ~20 °C</li> <li>• RH ~ 0</li> <li>• Temperature of mixed diluted exhaust ~ 20 °C</li> <li>• Secondary dilution ratio 70 - 100:1</li> </ul>
FEV	The temperature of the dilution air (for dilution tunnel and ejector dilution) is about 20 - 30 °C; the humidity of the dilution tunnel is not controlled. The humidity of the ejector dilution is controlled to 20 %.
Ford	Primary dilution: <ul style="list-style-type: none"> <li>• Temperature 108 F</li> <li>• RH T<sub>dew</sub> = 15 F =&gt; RH = 4 %</li> <li>• No hydrocarbon removal</li> </ul>
EPCD	<ul style="list-style-type: none"> <li>- temperature: 74 ± 4 °F</li> <li>- relative humidity 50 ± 6 %</li> <li>- dilution air provided from inside the test cell.</li> <li>- both temperature and humidity are controlled.</li> </ul>
UMN	<p>New Tunnel</p> <p>Primary Dilution:</p> <ul style="list-style-type: none"> <li>• Compressed air to 5 PSI</li> <li>• Controlled temperature: 25-150 °C</li> <li>• Controlled R.H. range from 10-70 %</li> </ul> <p>Similar capabilities with the secondary dilution</p> <p>Old Tunnel</p> <p>Primary Dilution</p> <ul style="list-style-type: none"> <li>• Compressed Dried Air with Silica Gel ~ 50 PSI</li> <li>• Ambient level temperature (Uncontrolled)</li> <li>• ~0 R.H.</li> </ul> <p>Similar characteristics at the secondary stage</p>

**11. What is the background level of particles in the dilution air? Is the dilution air filtered and if so how?**

Laboratory	Response
AEA	The dilution air is filtered through a Volkes Ltd VG2 panel filter (C96124/12/E) to remove dust then through a bank of activated charcoal filters to remove hydrocarbons and finally through another panel filter. The background level of particles in the filtered dilution air is not significant on particle counting.
Biel	Dilution air is filtered, filter unknown, original from AVL Smart Sampler II
EMPA	less than 300 particles/cm-3 counted by CNC dilution air filter in use
MTU	see attached table
FEV	The dilution air are filtered through a micro filter (filter class R, the separation grade: 99 %) and an active carbon filter. The background level is $< 2 \mu\text{g m}^{-3}$ .
Ford	Background 0.0042 $\mu\text{g/s}$ , typically, filtered air
EPCD	- Dilution air is filtered with a HEPA filter. - Background PM concentration is approximately 50 particles/cc.
UMN	New and Old Tunnel: HEPA Filters Background Concentrations is less than 1 Part./cc

**12. Is a method of hydrocarbon removal used for the primary/secondary dilution air?**

Laboratory	Response
AEA	Activated charcoal
Biel	No
EMPA	primary dilution air: hydrocarbon removal by activated carbon
MTU	Charcoal scrubber in primary
FEV	Primary dilution air is filtered by an activated carbon filter.
Ford	No
EPCD	No
UMN	New Tunnel: optional Old Tunnel: No

**13. What are the temperatures of the mixed diluted exhaust:**

Laboratory	Response
AEA	When testing a 2.7 l diesel vehicle, the temperature of the diluted exhaust in the primary tunnel rose from ~27 to 38 °C over an EUDC cycle
Biel	50 °C
EMPA	Chassis dyno: dependence on the operation mode, below 52 °C Engine dyno: dependence on the operation mode, below 100 °C secondary tunnel: below 52 °C
MTU	Primary: 46 °C ± 1 °C Secondary: ~ 20 °C
FEV	In the dilution tunnel the temperature is 20 - 52 °C; in the ejector dilution the temperature is 20 - 30 °C.
Ford	Temperature of mixed diluted air 96.5-97.5 F during typical FTP test with 3.8 l engine No secondary dilution
EPCD	Primary tunnel: approx. 200-300 °F Secondary tunnel: Only filter face temperature is recorded and is kept below 125 °F
UMN	New Tunnel: Close to adiabatic, depends on exhaust and dilution air temperature Old Tunnel: non adiabatic close to ambient ~25 °C

**14. What is the total residence time in the dilution system to the filters? What is the primary tunnel residence time? What is the secondary tunnel residence time (if applicable)?**

Laboratory	Response
AEA	The total residence time is ~ 0.45 s
Biel	The mixed residence time in the tunnel is ~1.5 s
EMPA	primary tunnel residence time: t ≈ 0.7-1.5 s
MTU	Tunnel residence time to filters = 1.34 s; time to particle size measurement point = 1.8 s
FEV	The residence time for the dilution tunnel is about 1.93 sec; the residence time for the ejector dilution is about 10 sec.
Ford	Tunnel residence time to filters = 1.5 s; time to particle size measurement point = 3.0 s
EPCD	Primary: ~ 0.75 s (with a 2000 cfm venturi)
UMN	New Tunnel: 0.1-8 sec between primary and secondary (variable optional) Old Tunnel: ~0.4 sec between primary and secondary Secondary to instrument for both tunnel is about 0.4 sec

**15. What is the residence time from the entrance to the dilution tunnel to the point where particulate size measurements are made?**

Laboratory	Response
AEA	~0.4 s
Biel	No known
EMPA	Chassis dyno: 3-5 s; Engine dyno: 2-4 s
MTU	~ 1.8 s
FEV	The residence time from the entrance of dilution tunnel to the point of the particle size measurement is 12 s.
Ford	~ 3 s
EPCD	No known
UMN	0.5-8.4 sec

**16. Is there any other stage of dilution prior to the entrance of the particle-sizing instrumentation that is not mentioned above (e.g. an ejector diluter mounted directly upstream of an EAA or SMPS)?**

Laboratory	Response
AEA	Not normally used
Biel	No other dilution
EMPA	Small ejector diluter (dilution factor: 10) Smart sampler (partial flow tunnel dilution unit)
MTU	No
FEV	No
Ford	No
EPCD	Ejector diluter mounted upstream of the SMPS
UMN	Ejector diluter is mounted upstream of SMPS or EAA (Dilution ratio from 4-100)

**17. Describe you method of particle size measurement.**

Laboratory	Response
AEA	<p>TSI SMPS  Dekati ELPI  QCM  TEOM  Andersen Mk III Cascade Impactor</p> <p>Details available in reports.</p>
Biel	<p>Scanning Mobility Particle Sizer  Manufacturer and model number:</p> <ul style="list-style-type: none"> <li>• TSI, model 3934 Includes Electrostatic classifier TSI Model 3071A and CNC model 3025A</li> <li>• Hydrocarbon removal: activated carbon (manufacturer: ETH Zurich/CH)</li> <li>• aethalometer</li> <li>• Photoemission, type PAS 2000 (EcoChem, GmbH Magnolienweg 1, Überlingen)</li> </ul>
EMPA	<p>SMPS/DMPS                      TSI  ELPI                                      Dekati</p> <ul style="list-style-type: none"> <li>• Calibration by means of PSL</li> </ul>
MTU	TSI Model 3030 EAA
FEV	<p>Following types of instruments are used:</p> <ul style="list-style-type: none"> <li>• Scanning Mobility Particle Sizer (SMPS) consists of a Condensation Particle Counter (CPC) Model 3010 and Electrostatic Classifier Model 3071A Manufacturer: TSI</li> <li>• Electrical Aerosol size Analyzer (EAA ) Model 3030 Manufacturer: TSI</li> <li>• Impactor (according by Prof. Berner) measured directly by the dilution tunnel.</li> </ul> <p>The ranges of the particle size are from 10 - 800 nm.</p> <p>The instruments are calibrated with PSL.</p>
Ford	*
EPCD	<p>Scanning Mobility Particle Sizer  Manufacturer and model number:</p> <ul style="list-style-type: none"> <li>• TSI, model 3934</li> </ul> <p>Nominal and actual range(s) of particle sizes:</p> <ul style="list-style-type: none"> <li>• We scan from 10 to about 500 nm.</li> </ul> <p>Method of calibrating particle size instrumentation (NaCl, PSL, other)</p> <ul style="list-style-type: none"> <li>• No method currently used, although voltage and flow readings have been checked before to ensure that particle sizes calculated are correct. Planning to implement calibration using PSLs.</li> </ul>
UMN	<p>CNC 3020 (TSI)  EAA 3030 (TSI)  SMPS similar to TSI 3934  CPC 310 (TSI)  CPC 3025 (TSI)  Size range from 7-1000 nm  Calibration method is done with the use of PSL</p>

**18. Have the particle losses of the sampling system up to the particle size measurement instrumentation been characterized, how?**

Laboratory	Response
AEA	Details are available in reports
Biel	No
EMPA	calculation of the diffusion losses, estimation of losses by thermophoresis in preparation
MTU	No
FEV	The particle losses are considered only by calculating particulate masses and by comparison of total particulate mass with impactor results.
Ford	No
EPCD	Losses calculated using sampling conditions and empirical equations found in the literature (such as Hinds' text)
UMN	Empirical Correlation taking into account the following particle losses: Brownian Diffusion Inertial impaction Turbulent deposition Sedimentation Thermophoresis