THE POSSIBILITIES FOR MEASUREMENT AND CHARACTERIZATION OF DIESEL ENGINE FINE PARTICLES – A REVIEW

by

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This review paper considers possible instrumentation for diesel engine fine particles exhaust emission evaluation. The modern diesel engines have extremely low particles emission almost at the level of measurement error of existing gravimetric measurement method. Since coarse particles are eliminated by new engine technologies, fine particles, with very negative effects on human health, dominate in the emission of current diesel engine. Therefore, it is necessary not only to measure mass of emitted particles but also to investigate other important particle characteristics as: particles number, particle size, particle number and mass distribution, particle active surface, particle composition, etc. Therefore, existing measurement technologies used in aerosol science can be used also to study diesel engine particles properties. This most common instrumentation in aerosol technique is shortly reviewed in the paper with special attention on candidate instruments included in EU program on portable emissions measurement systems.

Key words: *IC engine, diesel engine, exhaust emissions, measurement, particles emission*

Introduction

Particulate matter (PM) is the most important pollutant from diesel engine exhaust emissions with very adverse effects on human health. There is increasing evidence that several health effects are associated with the ultra-fine particles with diameters below 100 nm. Recent researches show that they can penetrate the cell membranes, enter into the blood, reach the brain, and even have some mutagen effects [1-3]. Therefore, exact measurement and control of diesel engine particles emission is an imperative. However, to understand what to control and measure, it is necessary to know essential properties of diesel engine particles [4].

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Particulate matter is composed both of coarse and fine particles [5]. Coarse particles are mainly eliminated in modern diesel engines (EURO 4 standard and later), so fine particles are the major problem. Fine particles can be found in two forms: as solid particles (which consist of elementary carbon, ash, metal traces, *etc*.) and as volatile particles compounds – organic (unburned hydrocarbons from fuel and oil) and non-organic (sulphur and nitrate compounds and other). Depending on temperature and other conditions, the volatile fraction may remain in the gas phase, condense on existing solid particles, and nucleate and form new liquid particles. Figure 1 roughly shows typical composition of engine particles [6]. Actually, the real composition varies depending on operating conditions, exhaust temperature and applied engine technology. Approximately, about 50% are solid particles, abut 30% are organic compounds, and about 20% are non-organic compounds. However, this composition varies and depends on applied engine technology. Old diesel engines (EURO 2) emitted more than 80% solid particles, but modern diesel engines (EURO 5) with diesel particulate filter (DPF) had less than 20% solid particles in whole particulate matter emission. Also, it should be kept in the mind that sample does not contain only particles formed in combustion process, but also secondary particles formed during cooling in exhaust and sampling lines.

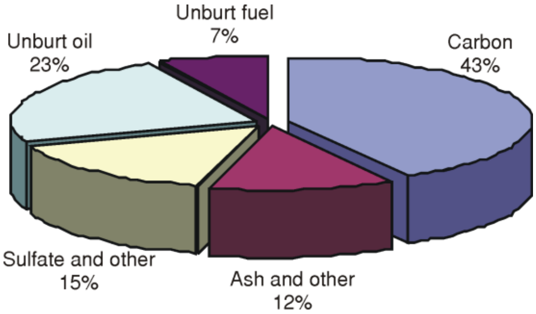

Figure 1. Possible composition of particles emitted from heavy-duty EURO 4 diesel engines

As concerns particles formation, there are several hypotheses, but of the theory Heywood is well accepted [7]. It says that particles are formed by the pyrolytic reaction in combustion chamber with disintegration of hydrocarbon chains under high temperature and without oxygen. During the hydrocarbon disintegration, the hydrogen content in a molecule is reduced and soot particles are formed with nuclei rich with carbon. All this is happening with growth of particle specific surface. After leaving combustion chamber newly formed particles are agglomerated (with possible oxidation) with further absorption and condensation. All other compounds like metals, organic and non-organic compounds from oil, ash from fuel and oil are absorbed on particle surface. Therefore, fine particles are distinguished by two particle formation phases, *i. e*. nucleation, when particle nucleus is formed, and agglomeration, when particles grow up.

Particles mass is mainly in the accumulation mode and their size is from 50 to 500 nm. This phase participates with 89-90% in total particle mass, but with only 10-30% in their total number. Particles in this mode are carbon nuclei with absorbed volatile organic compounds and polycyclic aromatic hydrocarbons, as well as non-organic sulphur and nitrogen compounds. The second mode is the so-called nucleation mode (mainly <50 nm) which has minor contribution in total mass but major in number. The particles in this mode origin mainly from liquid droplets formed by condensation of easy volatile organic and non-organic compound [5].

Particle size from these groups is less then 10 μm, and larger particles are appearing sporadically. Only particles smaller then 2.5 μm, *i. e*. fine particles, are present in the exhaust gas of new EURO 4 class diesel engines. The biggest interest is to detect and predict fine particles behaviour in diameter less then 100 nm (ultra fine particles) and 50 nm (nanoparticles) from nucleation mode, because it is suspected that they have toxic organic and non-organic components. However, the optimization of the engine combustion process and the application of aftertreatment devices not only reduces the emitted particle mass but also leads to significant changes in the nature of the particles, such as a relative increase in the volatile fraction. Therefore, besides the particle mass some other particle properties have to be considered, such as: particle size, particle size distribution, number of particles, particle surface, *etc*.

Depending of metric method (based on particle drag or inertia properties), particle size can be defined by aerodynamic diameter or mobility diameter [5]. Fine particles have an aerodynamic diameter less that 2.5 μm ($PM_{2.5}$). Ultra fine particles are from 5 to 100 nm, with approximate average diameter of 20 nm. However, fine and ultra fine particles have great specific surface, which make them excellent for accumulation of organic and in organic compounds. There is increasing evidence that several health effects are associated with the ultra fine particles with diameters below 100 nm [2]. For example, DPF had achieved dramatic reduction of particle mass emission (which is on detection limit for standard gravimetric method), with more than 95% reduction of solid particles. New problem are volatile fractions of ultra fine particles, which are found downstream of DPF and catalytic converters, although they almost do not have mass and their pathways are unpredictable and hardly could be modelled [8].

Figure 2 shows typical size distributions of diesel exhaust particles emitted from a heavy-duty engine equipped with a DPF [9]. Without DPF, most particles are in the size range of 30-300 nm (accumulation mode). After the DPF, the concentration of the accumulation mode particles is drastically reduced. However, a large number of particles occur in the nucleation mode. They dominate the particle number. Without DPF, volatile material condenses on the solid particles. As the trap removes the solid fraction, no surface to condense on is available. The resulting higher super saturation leads to homogeneous nucleation of the volatile material, passing the DPF in the gas phase. Both modes can well be approximated by lognormal (solid lines are lognormal fits to the measured data).

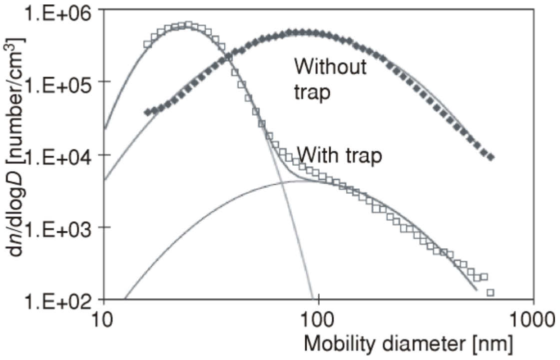

Figure 2. Particles size distribution of heave-duty diesel engine equipped with particle trap – diesel particle filter

A lot of attention has been focused on finding in correlations between particle number concentration and mass (which is on the limit of detection) [4, 10]. Direct correlation is not yet found because of the complex particle nature and because the sampling procedure can influence the measurement results. Actually, the complexity of the phenomena requires new measurement methods, which will increase knowledge on particulate matter phenomena. These methods are mostly used in aerosol science and can be included in future regulations. Therefore, they will be discussed shortly in the following text.

Methods for verification and research of particle emissions

The existing method to measure particle emissions for vehicle approval type test is the gravimetric method, with sample taken from an exhaust pipe through dilution tunnel. As can be seen from fig. 3 [11] the detection limit of this method is already achieved with the EURO 4 limit. Therefore, the sensitivity of gravimetric analysis has to be increased to remain applicable for low-emission engines for EURO 5 and EURO 6 limits. In addition, with the optimizations of the new generation engines with DPF and application of after treatment devices large amount of solid particles was eliminated, but prevailed relative increase of unstable volatile fraction. These fractions are composed mostly of unburned hydrocarbons and small amount of non-organic sulphur. Along with the insight that mainly very small particles are of importance for health effects, the question is raised whether mass is still appropriate metric for standard measurements of particle emission. The discussion indicates that the total mass probably is not a very good metric. However, it does not answer the question what is the best alternative.

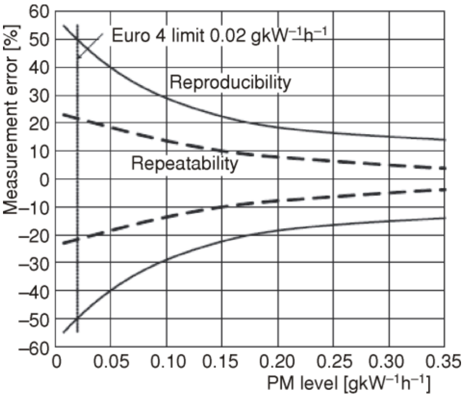

Figure 3. Reproducibility and repeatability of measurement results [11]

Of course, particles health effects are of the greatest importance. Recent results indicate that solid nanoparticles are of great importance. In the terms of inflammatory responses, a good correlation between health effects and particle surface is obtained [2]. Also, good correlation exist between active particle surface and solid particle number [10]. However, not much is known about effects of the volatile fraction [8]. Nevertheless, it is clear that lower particle concentrations will be a benefit for health, and therefore some requirements for new particles measurement methods can be concluded [9, 10, 12]:

- solid and volatile particle fraction should be considered separately,
- the solid fraction can be measured in terms of solid particle mass, number concentration, elemental carbon mass, and surface of the solid fraction,
- elemental carbon mass dominates for non-DPF emissions and is proportional to solid mass fraction; also, it has smaller health significance,
- as health effects depend on particle chemical composition, which is in good correlation with particle surface, it should be kept in the mind that total particles active surface increases with the increase of particles number; for the same particles mass, bigger particles number means bigger particles surface,
- the measurement resolution should be sufficient to distinguish nucleation from accumulation mode,
- due to the smooth shape of the size distribution no high-resolution measurement is required,
- nucleation is very sensitive to the way the exhaust is cooled and diluted; to obtain reproducible results this has to be considered carefully when designing the sampling system,
- to obtain representative results with respect to the typical operation conditions, the measurement method should be applicable for transient operation conditions,

- it is desirable to use the same metrics for engine emissions, occupational exposure, and ambient air,
- measurement method should have good repeatability (test-to-test variations) and reproducibility (lab-to-lab variations),
- there are highly developed instruments and good experience exist for measuring particle number concentrations using condensation particle counters (CPC), and
- an adequate calibration and verification of instruments should be of great importance.

Not all these requirements can be easily fulfil, and some compromise has to be found. It is clear that at least particles mass and number are important and should be measured. The advantage of the mass-based metrics compared to a measurement of the number concentration is that the mass is conserved during processes such as coagulation, until the number can be changed. For standard vehicle approval test, the focus is on the solid fraction only because these particles can be measured with higher repeatability. Volatile fractions can be removed by adequate sample heating or by using thermodesorber and possible condensation and nucleation can be avoided by adequate dilution techniques. This is the so-called "dry measurement" which has good repeatability and reproducibility but does not reflect "real life" conditions. "Wet measurement" is closer to "real-life" conditions because all potentially dangerous material is included in the measurement. However, in this case, the repeatability is much worse, but it can be interesting to study for research. In addition, size-resolved measurements should be used for research problems.

Therefore, the recommendation is to measure the solid fraction in terms of the number concentration for future type approval tests. This should be done in addition to the improved existing gravimetric mass measurement. This combination gives adequate importance to the very small particles without ignoring the total mass. A number of measurement systems have been developed or are under development for characterization of diesel particle emission. Any system has to include a sampling/preconditioning unit and the measurement system itself. Special problems are the calibration and verification of included instruments in a well designed sampling system [12].

To solve these problems and to propose future measurements methods, the UNECE WP29/GRPE group initiated the particle measurement program (PMP) in 2001. The aim of PMP is to make recommendations for a new or additional particulate matter measurement system to be used for type approval testing and corresponding emission standards for both light and heavy-duty engines. After the initial assumptions [5], GRPE/PMP group of experts studied possibilities of different measurement methods of fine particles emission control and defined proposal of installation and sampling procedure that is included in engine emission regulations [13]. Figure 4 shows a schematic of the configuration for the measurement of particle mass and solid number concentration. According to this proposal, standard constant volume sampling (CVS) dilution tunnel can be used. The exhaust gas sample, taken from the tunnel by particle sampling probe (PSP), goes to cyclone pre-classifier (PCF), witch removes coarse particles. After PCF one part of sample goes to improved gravimetric measurement of particle mass, and another is going via particle transfer tube (PTT) to volatile particle remover (VPR) for particles number measurement. In VPR, after dilution with hot air (PND1), the sample is driven through heated evaporation tube (ET), heated up to 150 ^{o}C. As sample has to be cold at entrance of particle counter, and to prevent condensation, the sample is cold diluted (PND2) and then it is driven to particle number counter (PNC). Work with such installation has shown good repeatability and reproducibility [14].

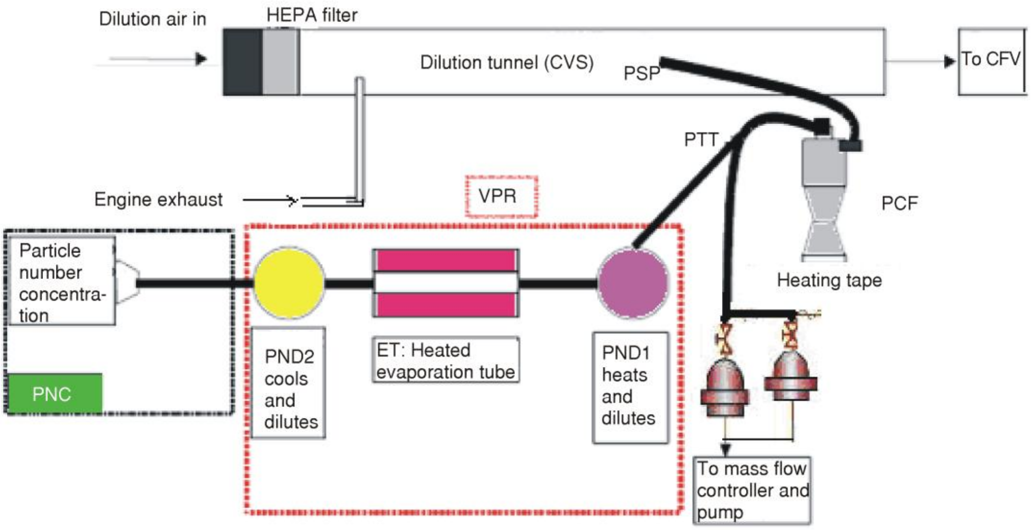

Figure 4. PMP set-up of laboratory instruments for particle number measurement

Basic elements of previously described installation are:

Improved particulate mass measurement includes cyclone pre-classifier which remove particles bigger then 2.5 μm. Then, the sample is passing through heated line where its temperature is kept bellow 52 °C for more than 0.2 s. Heated line leads the sample to primary or secondary filter holder. Filter face velocity is kept between 50 and 80 cm/s and it is recommended to use Pallflex TH40 filter with no backup. Flow controller can be installed at the filter outlet.

Dilution system is very important. The design of the sampling and dilution system determines largely what will be measured later. It serves to (a) reduce the concentration in the raw exhaust to a concentration which can be handled by the measurement system, (b) reduce the temperature to an adequate value, and (c) control the condensation and nucleation processes mentioned above in the desired way. The third item is important for particle number measurement.

Cooling the exhaust with no or moderate dilution will cause a condensation. Most volatile material will then be found in the nucleation phase. On the other hand, a sufficient dilution at increased temperature where the volatile components are still in the gas phase will prevent nucleation (it is called hot dilution – PND1). Other volatiles in liquid phase or condensed on solid particles can be removed by heating the sample over 150 °C. As the sample has to be cold at the entrance of particle counter, and to prevent condensation, the sample should be again diluted (it is called cold dilution). Only solid particles are measured in this case.

So far, *CVS* or *constant volume sampling dilution* principle is used for regulated emission measurements. Such dilution is enough for gravimetric measurement of particle mass. However, for particle number measurement additional hot and cold dilution should be added to enable particle counting and to prevent condensation. These additional diluters have to be simple and to enable high dilution ratio. The actual dilution ratio should be determined by simultaneous measurement of CO_2 concentration up- and down-stream of dilution system during the test. The simplest way of dilution is to introduce the dilution air just downstream of the sampling probe before the heated partial flow mini dilution tunnel, where it mixes and dilutes the exhaust sample. However, there are different types of additional diluters, but mainly two of them are frequently used: ejector diluter and rotating disk diluter.

Ejector diluter (fig. 5) is based on the function of a Venturi nozzle. The operation principle of ejector diluter is based on under pressure caused by the dilution gas flowing through an annular nozzle. If the dilution gas flow is increased the under pressure increases correspondingly and the sample flow is increased. Thus, the dilution ratio remains approximately constant, if the pressure in exhaust pipe is constant. The dilution factor is typically approximately 10. For higher dilution ratios, several diluters can easily be cascaded. The dilution ratio depends on the inlet pressure. Diluter and dilution air can also be heated to keep sample temperature. Normally, first stage diluter is heated and second is cold.

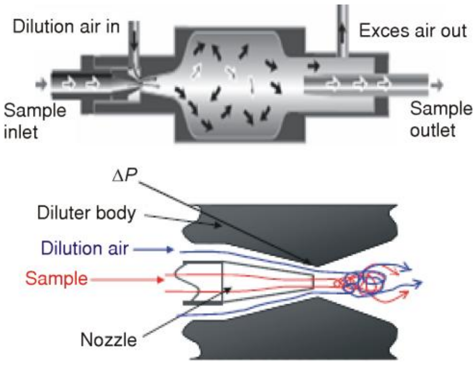

Figure 5. Principle of ejector-diluter [15]

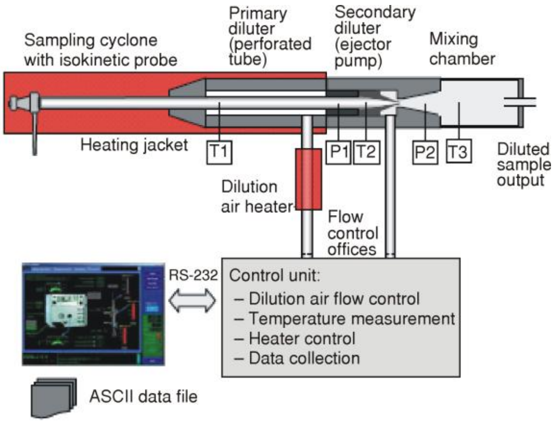

Figure 6. Fine particle sampler

Ejector diluter is successfully applied in Dekati fine particle sampler (FPS) (fig. 6) [16, 17]. It is a sampling and dilution device with possibilities to control dilution ratio, temperature and other parameters during research tests. Compressed dilution air is used to create sample suction in a conical nozzle. Sample flow depends on the pressure drop across the ejector, which is measured by two pressure sensors (P1 and P2). First dilution stage is located before the ejector and dilution air (heated to avoid volatile material condensation) is introduced into the sample flow through perforated tube. Dilution ratio of the FPS system can be selected between 1:15 and 1:200, probe and dilution air can be heated up to 400 °C.

According to GRPE/PMP recommendation for sample conditioning during particle number approval test (fig. 4), the above-mentioned company has developed Dekati engine exhaust diluter (DEED) [18]. This device uses two Dekati ejector diluters [15] connected with evaporation chamber. First diluter dilutes the sample with heated dilution above 150 °C. Then the sample is heated in evaporation chamber to a temperature above 300 °C to evaporate all volatile particles. After thermal conditioning, the sample is again diluted at 20 °C, which reduces the particle concentration along with the temperature to a suitable level for the CPC. Overall dilution ratio of the instrument can be selected as 100 or 1000.

Rotary disk diluter is product of companies Matter Engineering [19] and TSI Incorporated [20]. It is more complex, but can provide hot dilution and high dilution ratio. It can reach high dilution ratio up to 1/3000. It works well for particles below 1 μm; losses by impaction cause errors for larger particles. Figure 7 illustrates the principle of the dilution method. There are two separate gas channels: the raw gas channel and the diluted measurement channel. Small volumes are transported from the raw gas channel to the measurement channel by cavities in a rotating disk. The ratio of dilution of raw gas is a linear function of the cavity volume, the number of cavities on the disk, the frequency of rotation, and the flow in the diluted gas channel. The flow rate of the diluted gas is restricted to a few liters per minute.

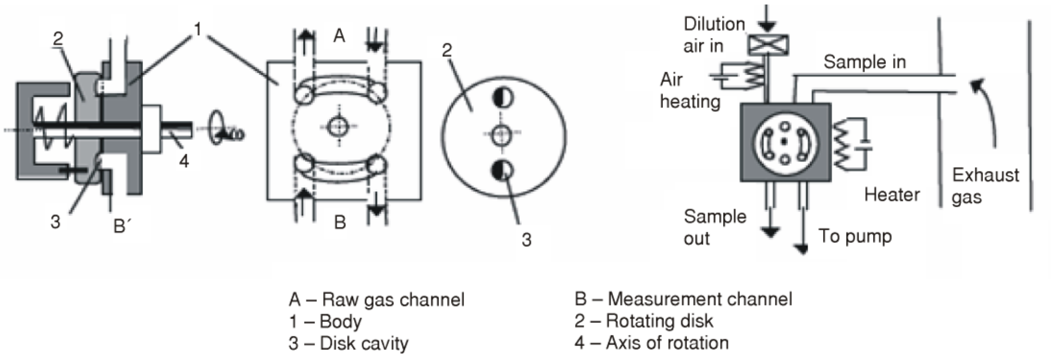

Figure 7. Rotary disk diluter

Volatile particle remover is a very important device, which has the role to eliminate evaporative liquid droplets, mostly composed from large hydrocarbons, and volatile organic compounds, which are condensed in exhaust gas flow during expansion and sampling. Their occurrence is very uncertain, undefined and depends on sampling condition and applied engine technology; so they can affect reproducibility and repetition of measured data. The example of volatile particle remover is the DEED.

Every attempt of warm up diluted exhaust gas could eliminate in certain extent condensed components. The simplest way is to use long enough evaporation tube heated over 150 °C, which enable evaporation of volatile compounds and keep them in gas phase. More complex is so called thermodenuder (or thermodesorber), which is a frequently used instrument in aerosol technique for removal of volatile components by heating them up to 250 °C and absorbing them in charcoal [21-23]. Its drawback is that some solid particles can be also lost and this loss is taken in account by manufacturer recommendation.

Figure 8 presents schematic of thermodesorber and fig. 9 [10] shows particles concentration before and after thermodesorber (TD). After heating, the sample enters the absorber section, where the vaporized compounds are adsorbed in active charcoal. We may see very good efficiency of removing condensed compounds from nucleation phase. Unfortunately, instrument may remove some solid particles from accumulation phase. Therefore, simple heated evaporation tube is better solution, but needs additional cold diluter downstream to prevent condensation and to enable optimal temperature before particle counter.

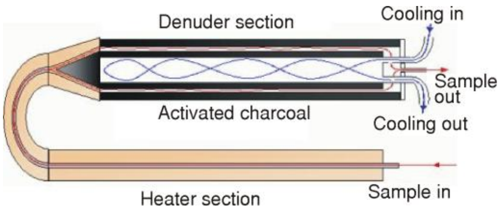

Figure 8. Schematic of thermodesorber

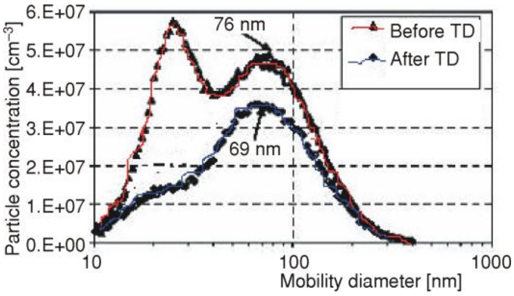

Figure 9. Particles distribution before and after thermodesorber

Particle number counter (PNC). Particles in the "soot-size" range are in general too small for direct optical detection methods. Therefore, aerosol particles are first enlarged inside the CPC by surface condensation of supersaturated n-butyl alcohol. The enlarged particles are counted using a simple optical detector. The CPC is an optical counter that has been very widely used as an aerosol technique for concentration determination [24].

Its working principle is shown in fig. 10. Particles from diluted exhaust gas sample are driven in contact with easy evaporable butyl alcohol, where super saturation occurs by cooling the sample, so the condensation of alcohol on particle surface increases the particle size enabling an optical particle counting. Particles grow until they reach range of 10 μm when optical detection is possible. Needed super saturation is increased with reducing particle diameter, what limits detection of very small particles (smaller then 10 nm). In addition, recommended particle maximum number is 10000 particles/cm^3. Near this limit particles overlap during their pass across optical beam and particle number detection is not accurate.

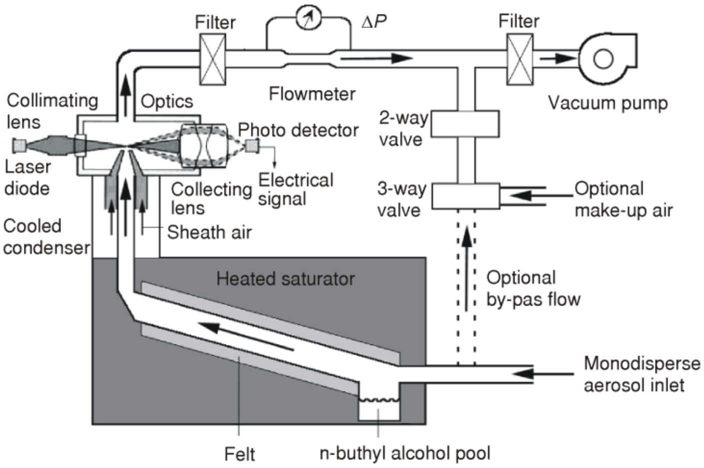

Figure 10. Condensation particle counter

After the acceptance of the PMP proposal for EURO 5 engine particle emission measurement, special instrumentation for particle number evaluation in vehicle emission has been commercially available. Company TSI Incorporated has developed engine exhaust condensation particle counter (EECPC) [25] which also incorporates hot and cold rotary disk thermodiluters, that dilutes the sample up to 1000:1, and thermal conditioner air supply, which heats the sample up to 400 °C [20]. Also, company GRIMM Aerosol has developed PMP-Automotive condensation particle counter in compliance with GRPE/PMP for EURO 5 Regulation 83 [26]. There are also other commercially available particle counters.

Finally, one more interesting CPC instrument will be mentioned. Company TSI has developed water based CPC, which uses water instead of butyl alcohol [27]. In the instrument, water is inserted in the sample through a tube made from porous media. After the saturation in the tube, the particles grow and go in the optical particle detector.

The results of measurements of particle concentration in the exhaust of light and heavy-duty vehicles, according to GRPE/PMP test proposal, which includes described procedure, devices and instrumentation, have been well presented in final reports of PMP inter-laboratory correlation exercise [13, 28]. The CPC is also effectively used in conjunction with other instruments for measurement of other particle properties, especially particles size distribution. These other instruments are mainly used in research and scientific projects on aerosol problems with the aim to understand better particle characteristics. They will be also shortly discussed.

Cascade gravimetric impactor measures particle mass distribution. It uses aero dynamical particle properties for their separation. After acceleration and sharp declination, small particles follow exhaust gas, but the bigger ones drop on the impactor stages (aluminum foils). Impactor stages were designed in cascade for separation between particle sizes. There are 4 to 12 stages for measurement mass size. Every stage is for one mass size represented by their average aerodynamic diameter (fig. 11) [29, 30].

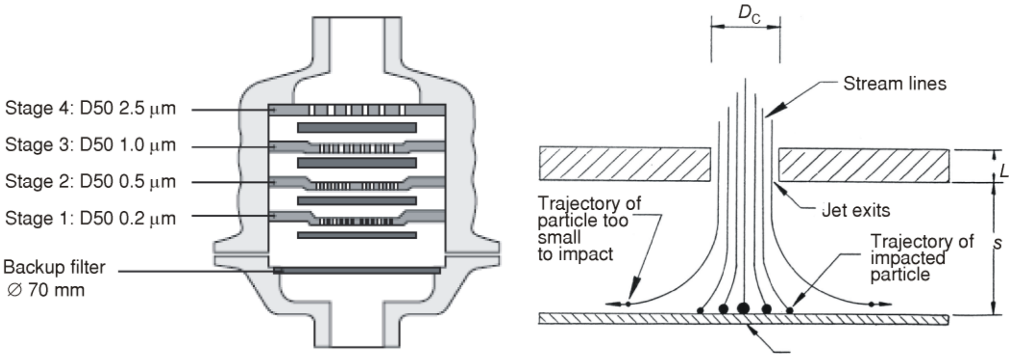

Figure 11. Cascade gravimetric impactor design (left) and principle of operation (right)

Gas flow is accelerated in delivery nozzle of each stage, and it is enforced to go around over each cascade stage. First nozzle plate has small number of large orifices (D_c, fig. 11) and then number of smaller orifices increases, so the steam velocity increases in each stage. The distance between stages is chosen to keep velocity high enough to prevent deposition of smaller particles. Therefore, large particles settle in first stages and smaller at latter. After settling of particles on impactor stages, stages are measured gravimetrically, so particle mass distribution by particle size, represented by its aerodynamic diameter, can be obtained. If necessary, the deposit can be chemically analyzed. Disadvantage of this instrument is that it cannot provide a high temporal resolution but it can provide an integrated value over a transient cycle.

The electrical low pressure impactor (ELPI, Dekati Ltd.) is designed to measure size distribution of aerosol particles in the size range from 0.03 to 10 μm aerodynamic diameter (fig. 12) [16].

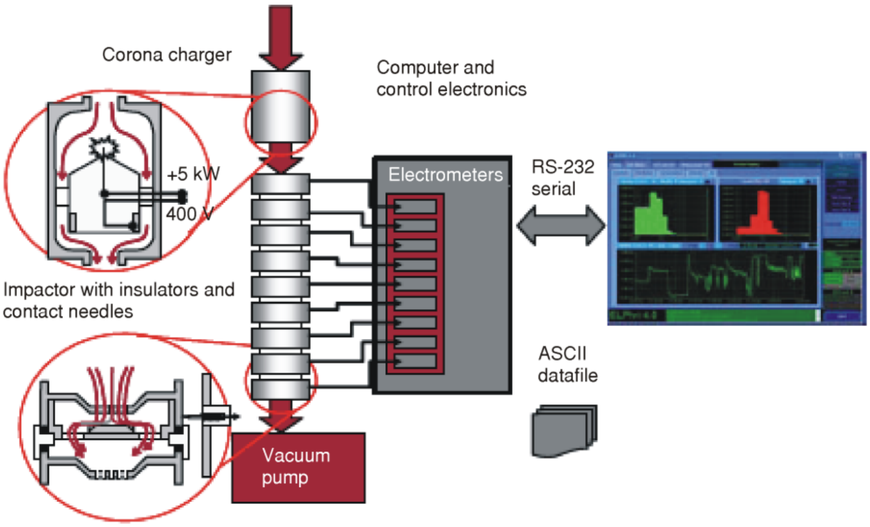

Figure 12. Electrical low pressure impactor

ELPI has similar design as cascade digital gravimetrical impactor, but main advantage is near real-time measurement of size distribution and concentrations. It has 12 stages. First stage is for large particles till 7 μm, and last stage is for the smallest particles with diameter of 30 nm. Sampling rate is 1 Hz. Size distribution, measured by ELPI, is defined with aerodynamic diameter and to each cascade plate corresponds relevant aerodynamic diameter [31, 32].

Instrument operating principle is based on the fact that, after acceleration and sharp declination, small particles pass a cascade and heavier ones settle on cascade plate. The ELPI operating principle can be divided in three major parts: particle charging, size classification in a cascade impactor and electrical detection with sensitive electrometers. The particles are first charged in the corona charger. After charging, the particles enter a cascade low pressure impactor with 12 electrically insulated collection stages. The particles are collected in the different impactor stages according to their aerodynamic diameter, and the electric charge carried by particles in each impactor stage is measured in real time by sensitive electrometers. This measured current signal is directly proportional to particle number concentration and size. Measured current signals are converted to particle size distribution using particle size dependent relations. The result is particle number concentration and size distribution.

Recently, company Dekati Ltd. has introduced improved version of this instrument [33]. This ELPI Plus impactor has 14 stages enabling measurement in the size range of 6 nm to 10 μm with 10 Hz sampling rate.

Differential mobility analyzer (DMA) is an instrument using as operating parameter electrical mobility diameter). A number of DMA designs have been developed and presented in the literature [34]. They can be made as: single-channel DMA or multi-channel DMA, as well as for fine or nano particles.

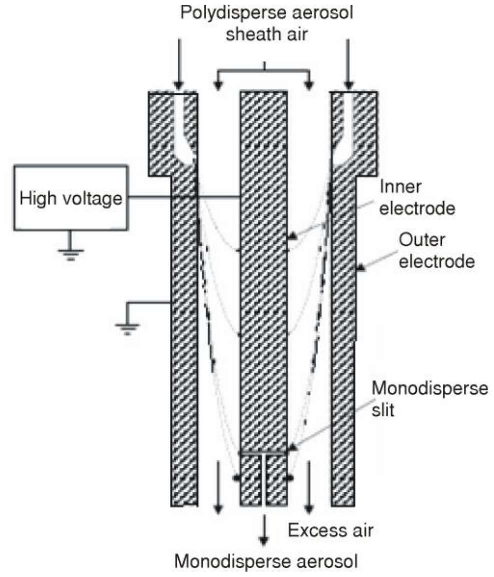

Figure 13. Differential mobility analyzer

Single-channel instrument (fig. 13) is operating in following way: before entering in classificator, particles in diluted sample of exhaust gas get bipolar charge by krypton radiation. In central part of the instrument is an electrode connected in high voltage circuit. Smaller particles under the stronger influence of electro magnetic field are attached on electrode, but the bigger ones slip under their inertia in the analyzer by air flow which exits the analyzer at the bottom. Downstream on the electrode is a narrow hole through which flow only particles with the relevant diameter. This means that only particles with same charge and with the almost same size diameter are drawn by electro magnetic force in orifice in the electrode. Particles of unique size (mono disperse aerosol) exit at the end from analyzer electrode and enter in particle counter.

Scanning mobility particle sizer (SMPS) is a combination of two instruments (fig. 14): differential mobility analyzer (DMA) and CPC [35]. By varying the central rod voltage of the DMA, particles of different electrical mobility are sent to the CPC where their number concentration is measured. Combining this information with the mean mobility of the particles determined by the potential on the central rod of the DMA at every scanning step, the size distribution of the sample aerosol can be determined. This combination allows to obtain particle size distribution and concentration. In this combination, DMA continually

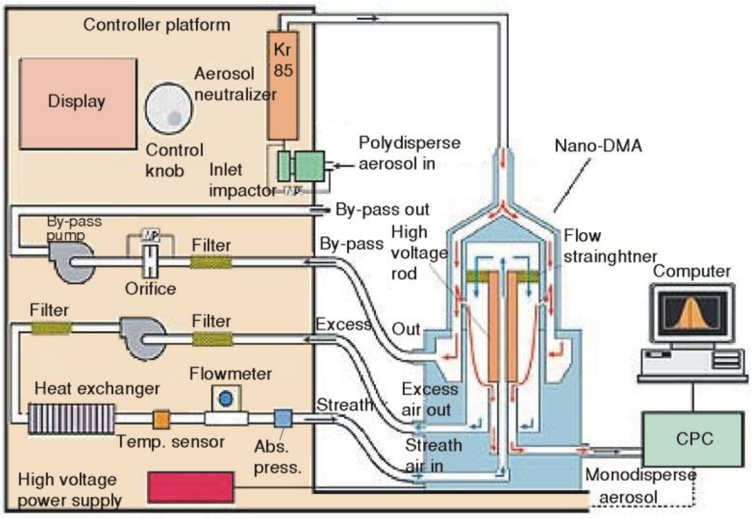

Figure 14. Scanning mobility particle sizer

changes voltage that gives at the DMA outlet mono-disperse gas with different size of particle for further concentration analyze. It is important to know that this set-up is not convenient for continual measurement, because one scans needs approximately a couple of minutes.

Very detailed analysis of different mobility spectrometers is presented in [36, 37]. Interesting combination of previously mentioned working principles is ***electrical mobility spectrometer*** (EMS) [38]. It is useful for continual measurement of particle size distribution and its design remind on ELPI, but here particles are affected by electro magnetic field, so this instrument is operating with electrical mobility diameter. Its principle of operation is shown in fig. 15. In the EMS, aerosol sample pass through a charger that sets a charge on the particles and enter a classifier separately but together with sheath air flow through an annular and exit. Electric field is applied between the inner and outer electrodes and forces charged particles having specific mobility to deposit on a designated electrode ring. An array of electrode rings is connected to a series of sensitive electrometers where electrical current signals are detected and translated in size distributions.

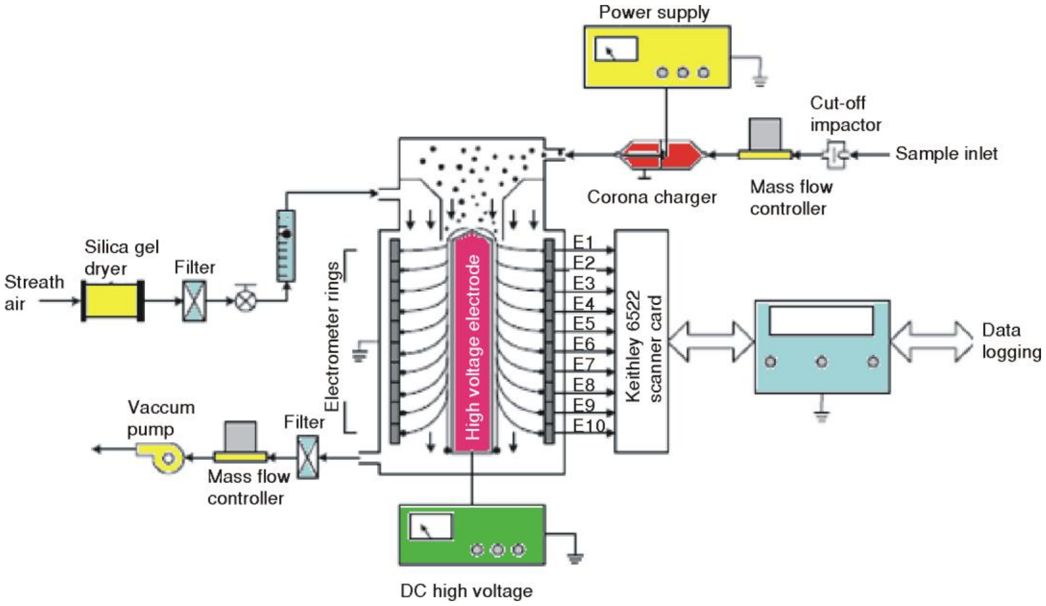

Figure 15. Electrical mobility spectrometer

Special design of previous spectrometer is ***Engine exhaust particle sizer*** (EEPS) from the TSI Incorporated company (fig. 16) [39]. This instrument gives continuously particle size distribution based on electrical mobility diameter. It draws a sample of the exhaust flow in the inlet where particles are positively charged using a corona charger. Charged particles are then introduced to the measurement region near the center of a high-voltage electrode column and transported down the column surrounded by sheath air. A positive voltage is applied to the central electrode and creates an electric field that repels the positively charged particles outward according to their electrical mobility. Charged particles strike the respective electrometers and transfer their charge. A particle with higher electrical mobility strikes an electrometer near the top; whereas, a particle with lower electrical mobility strikes an

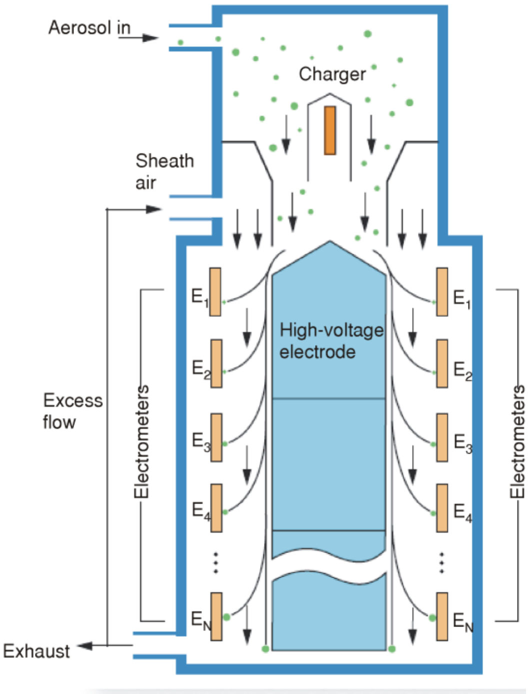

Figure 16. Engine exhaust particle sizer

electrometer lower in the stack. This multiple detector arrangement using highly sensitive electrometers allows for simultaneous concentration measurements of multiple particle sizes.

Diffusion charger (DC) can be effectively used to monitor the particle surface concentration, or combined with a CPC to derive the mean surface diameter [40]. It is a relatively simple and compact instrument with good time resolution below 1 s. The operating principle of this instrument is based on diffusional charging of the particles, followed by a current measurement caused by the trapped particles in a Faraday cage. The current signal is proportional to a particle dimension that is between length and surface area of the particles. The diffusion charging electrometer (DCE) LQ1-DC from Matter Engineering [41] was calibrated to active surface area, and the electrical aerosol detector (EAD) from TSI [42] was calibrated to particle length.

Figure 17 shows the operating principle of the TSI EAD [41], which is based on diffusion charging of particles, followed by detection of the aerosol via a sensitive electrometer. After the aerosols have entered the instrument, the flow is split and the part pass through a filter and corona ionizer, and the remaining to the diffusion charger. The flows

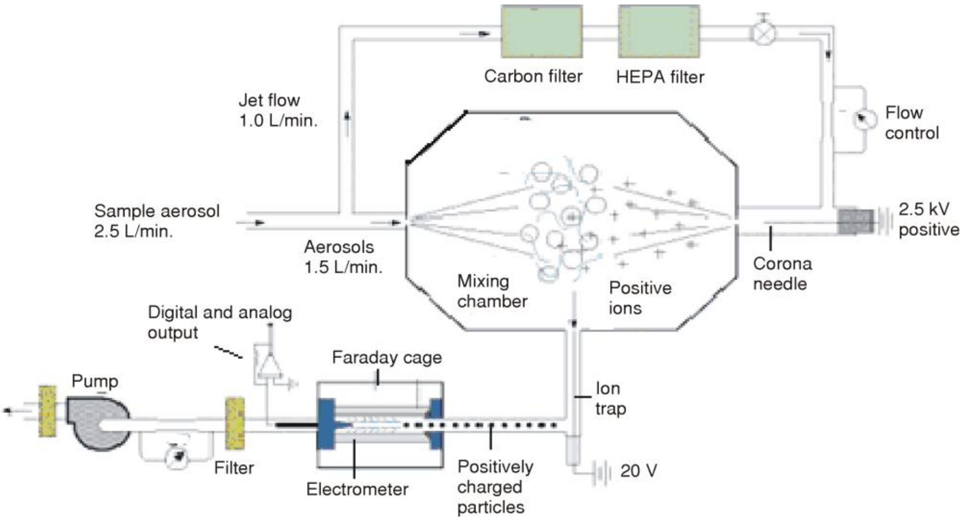

Figure 17. Working principle of TSI electrical aerosol detector

reunite in a mixing chamber where particles from the main aerosol flow mix with the ions coming from corona charger. This mixing brings the aerosol particles in a defined charge state. The charged aerosol then passes through an ion trap to remove excess ions and moves on a sensitive aerosol electrometer for charge measurement. The measured current is proportional to the mean aerosol diameter concentration. The aerosol diameter concentration (in mm/cm^3), also called total aerosol length, is defined as the length of a chain with all particles from 1 cm^3 of measured aerosol lined up on the chain.

Portable emissions measurement systems

So far, engine exhaust emissions are controlled in test laboratories with standard measurement equipment, using standard protocols and test cycles. In such operating conditions, modern diesel engines, using new technologies, have extremely low exhaust emissions, almost at the level of ambient air. New technologies for extra low engine emissions put new question: does emission testing in standard laboratory corresponds to real life emission of vehicle in-use? To answer this question it is necessary to measure vehicle in-use emission. However, such testing is very complex and still there is not reliable and appropriate portable emission measurement system (PEMS) that can be used in real-service vehicle operation.

The European Union is currently undertaking steps to check the in-use exhaust emissions of heavy-duty diesel vehicles. The European Commission launched in January 2004 a co-operative research program (the EU-PEMS Portable Emissions Measurement Systems Project) involving the European heavy-duty engine manufacturers and PEMS suppliers [43]. The PEMS Pilot Program shall facilitate the introduction into the European heavy-duty vehicle emissions legislation the use of PEMS as a tool for in-service conformity (ISC). The application of PEMS is ranging from large heavy duty engines to the smallest light-duty vehicles and more recently to off-road mobile machinery. The current commercially available PEMS for gaseous exhaust measurements exhibit measurement performances that are close to the ones of laboratory grade systems. While instrumentation and the test methods are mature for gaseous emissions, the development of portable PM instruments for PM measurement and their test protocols remain a complex challenge. EU PEMS program has to solve all these problems.

As already discussed, a variety of aerosol instrumentation is available for particle number and size measurements: SMPS, CPC, ELPI, EEPS, and DC. The main scope of the EU PEMS program is to validate the use of these instruments for ISC of heavy-duty engines and to evaluate the test protocol, which can be implemented in the European type-approval legislation. The current work has to evaluate the measurement performance of the candidate portable systems compared to a standard method and also to check their sensitivity for the future low PM emissions standards. The on board application of the standard PM measurement principles is challenging as PEMS are required to be small, lightweight, and easy to install, to be simple to operate, to work with low power consumption, to have good sensitivity for low PM concentrations, and to be affordable to purchase.

Very good review of potential measuring systems is presented in [43-46] including the most important results of comparative testing of different available instruments. Currently available portable candidate systems and their principles of operation will be discussed shortly. Actually, there are two different candidate systems. The instruments that require a sampling system and only sample a portion of the exhaust and the instruments that require no dilution and are fully immersed in the exhaust flow.

The instruments that require a sampling system

The AVL ***micro soot sensor*** (MSS) [47, 48] is a system for continuous measurement of soot (not total PM) concentration in diluted exhaust from IC engines. The device is sensitive for soot (detection limit ~5 μg/m³) with a low interference from other components. With the additional option "Conditioning unit" measurements upstream a DPF are also possible to evaluate the efficiency of the DPF.

Figure 18 shows its working principle. Highly absorbent soot particles are irradiated with modulated light. The periodic warming and cooling and the thus-resultant expansion and contraction of the carrier gas can be viewed as a sound wave and detected with microphones. Clean air does not produce a signal. With soot-laden air or exhaust gas, the signal increases proportionately with the concentration of soot in the measured volume.

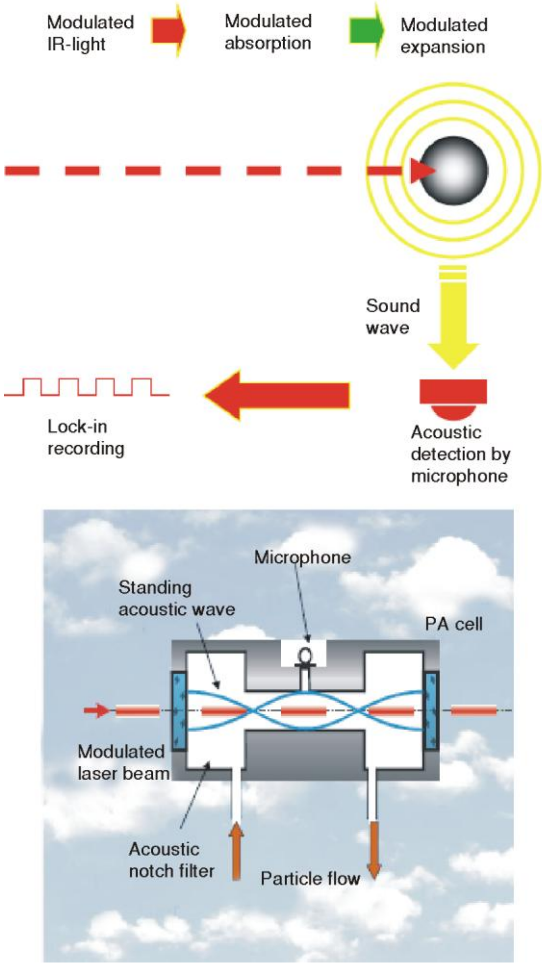

Figure 18. Operation principle (top) and schematic (down) of AVL micro soot sensor

The photoacoustic (PA) measuring principle [49] restricts the input pressure of the measuring chamber of the AVL MSS to the ambient pressure ±50 mbar. The temperature of the exhaust gas passing through the measuring chamber of the AVL MSS may not exceed 60 °C. To enable the work at high exhaust gas temperature and pressure, additional control unit is conditioning the exhaust sample. In the event of insufficiently diluted exhaust gas there is the risk of the formation of condensate in the measuring chamber of the AVL MSS. In order to avoid condensate formation, sufficient dilution of the exhaust gas should be provided for. With the dilution module of the optional exhaust gas conditioner, dilution of the exhaust gas to a dilution rate range of 2 to 20 is possible. No correction for losses or additional MSS calibration was needed when using its exhaust conditioning unit.

A picture of the portable ***control system m-PSS*** (Micro-PSS) is shown in fig. 19 [50]. The m-PSS is a particulate sampling system based on partial flow method and variable dilution. The partial flow sampling method enables to use the m-PSS with engines of any size, from small ones to big naval units. It uses a

47 mm filter and collects PM with a filter face velocity of 43 cm/s at a filter temperature of 44 ± 1 ºC [51]. The device satisfies the specifications for evaluating the particulate in transient cycles. m-PSS is a stand-alone unit; the overall weight is less than 35 kg thus allowing the installation on running vehicles for in-use compliance tests. The system needs 24 V DC power supply and it is ready to run in a very short time. m-PSS is provided with low inertia mechanical flow-meters connected to variable-speed volumetric pumps.

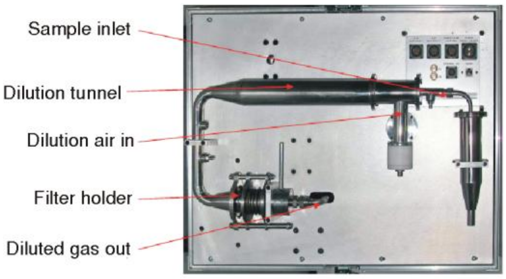

Figure 19. Control systems m-PSS

This configuration allows to have a very low response time in transient conditions. Thank to this the system can be used with the "on line" control method thus avoiding to run pre-tests. The mechanical-type flow meters offer high stability and repeatability, so that calibration procedures need to be carried out only after long time from each other. Additionally (not shown in fig. 19) m-PSS can be equipped with a real time particulate sensor that operates by electrostatic charging of PM particles [51]. Once integrated and corrected with gravimetric measurement this signal can be used to determine the quantity of particulate collected on the filter second per second.

The Horiba ***on-board transient response particulate measurement*** (OBS-TRPM) system includes two particulate measurement systems: partial flow sampling system (PFSS), and diffusion charge sensor (DCS) [45, 52]. Figure 20 shows the schematic and operation principle of this instrument. The left side of the figure shows a standard PFSS, which measures gravimetric particulate mass on a filter base. The dilution air is introduced just downstream of the probe before the heated mini dilution tunnel. After mixing in the tunnel, the sample, kept at 47 °C, passes a cyclone with cut point at 6 μm and then goes to 47 mm filter holder and, eventually a small amount of this diluted exhaust is bypassed to the diffusion charge sensor DCS module for real time measurement, shown at the right side of the figure. This flow is measured by an orifice in the FO module. Actually, DCS of OBS-TRPM instrument uses TSI electric aerosol detector (EAD) previously described [42]. If necassary,

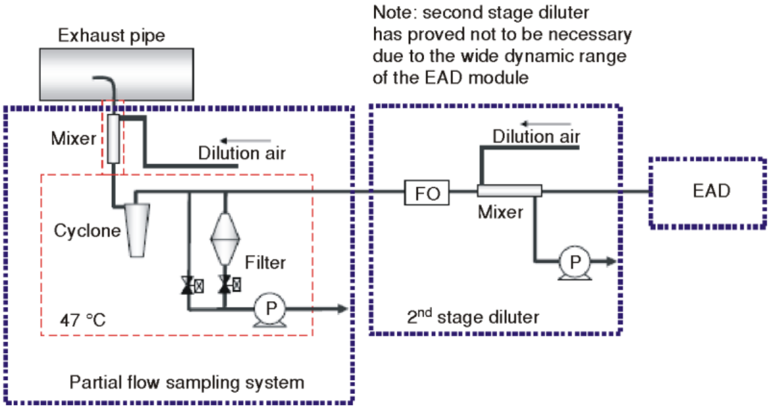

Figure 20. Horiba OBS-TRPM

second diluter can be installed in front of EAD. TSI EAD (a DCS) is real time particle instrument, which measures the particle concentration on a second by second basis, and at the same time PFSS collects PM on a gravimetric filter. The filter weight gain is used to provide a calibration constant to the real time particle signal.

The ***sensors' emissions measurement technology*** (SEMTECH), product portable particulate mass measuring device **(**PPMD**)** (fig. 21) is based on the quartz crystal micro-balance (QCM) technique for PM mass measurements. The portable system directly measures the mass of exhaust particulates under real world conditions, with nano-gram sensitivity. The unit integrates three key, patented subsystems from the SEMTECH product line, into a single self-contained unit: the exhaust flow meter (EFM), the micro proportional sample system (MPS), and the carousel quartz crystal micro balance (CQCM). When used in conjunction with the SEMTECH-DS, Sensors' gaseous emissions analyzer, it can enable complete measurement compliance with the EPA's testing requirements [53].

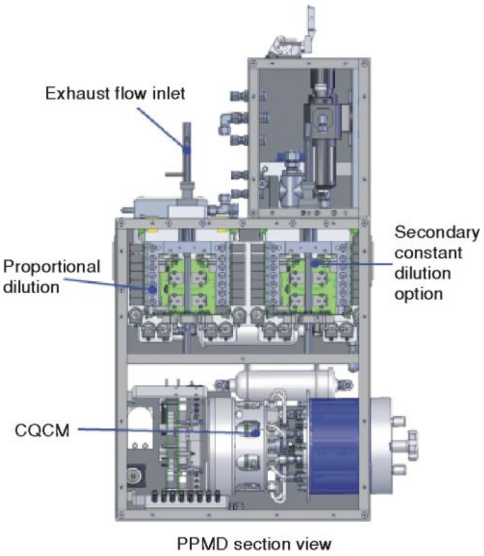

Figure 21. Sensors' portable particulate mass measuring device

The SEMTECH-EFM is designed to measure total raw exhaust flow. It uses an averaging pitot tube that operates under the Bernoulli principle.

The SEMTECH-MPS is a partial flow dilution system that controls the dilution air flow with a multi-bit parallel solenoid array. Each solenoid is fitted with a critical flow orifice to provide precise and repeatable flow characteristics. The system uses a signal from the EFM to establish a diluted flow that is proportional to the total raw exhaust flow. This patented technology operates at 10 Hz, providing a near instantaneous adjustment of sample flow. Beside this initial proportional dilution, secondary constant dilution can be applied if necessary.

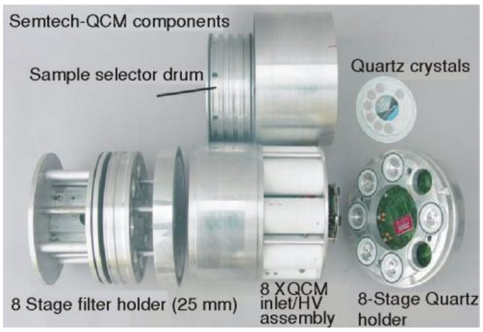

Figure 22. Quartz cristal microbalance

The SEMTECH-CQCM (fig. 22) is comprised of eight crystals, which oscillate at about 10 kHz. As the diluted sample from the MPS is pumped past the crystals, a corona discharge generates an electrostatic force, driving the particulates to collect on the crystal surface. As the particulates accumulate, the natural frequency of the crystal drops in relation to the mass. When one crystal is done sampling, the system automatically changes to the next available crystal, allowing each one time to stabilize before sampling again. In this manner, the system can operate unattended for up to eight hours [54].

Based on the well-known ELPI™ technology, the ***Dekati DMM*** is an instrument which measures the mass concentration of airborne particles in real time by combining aerodynamic and mobility size particle classification [55, 56]. The instrument involves a corona charger with mobility analyzer stage, and a six stage inertia impactor with electrical detection (fig. 23). A diffusion charger is used to give precisely determined charge to all measured particles. Charge level is close to saturation charge level for each particle size. After the charging region and before the impactor stage, a static electrical field is used to deflect smallest particles (almost without inertia and aerodynamic properties) to the charger mobility electrode; an electrometer is used to measure this current. In this section the smallest particles are defined by their mobility diameter. The particle size classification is accomplished in a 6-stage inertial impactor where the particles are separated by their aerodynamic properties in a low-pressure impactor with insulated collection electrodes. Sensitive electrometers are connected to impactor collection sensors and particles impacting to a corresponding collection plate generate an electrical current for that electrometer. This current is proportional to the amount of particles in each size range. Combining the particle mobility size information from the charger and aerodynamic size from the impactor enables calculation of the effective density of the particles required for conversion from measured current values to particle mass concentration. Particle size range is from 0 to 1.2 μm and total mass concentration range is from 1 to 1000 μg/m^3, even higher for short periods of time. Time resolution is 2-3 seconds.

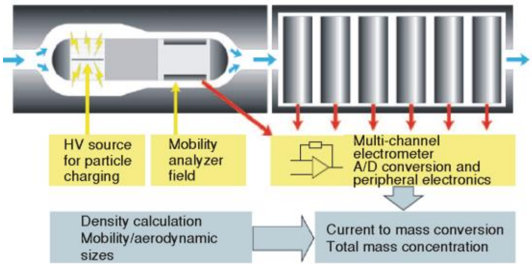

Figure 23. Dekati DMM

The instruments that do not require a sampling system

These systems are very promising because they are relatively simple, small in mass and weigh, do not need special dilution and ease for installation.

The Dekati ***electrical tailpipe PM sensor*** (ETaPS) is based on electrostatic principles for particle detection in real time [57, 58]. This is a new-sensor type device for in-situ tailpipe PM measurements. It does not require sampling or dilution, but needs sheath air. Figure 24 shows its working principle. When exhaust flow passes through the inner charging chamber a certain amount of charge is attached to all solid and volatile particles. The charge carried by particles leaving the outer charging cage are then measured with a sensitive electrometer (it is so-called escaping charge technology – ECT): the amount of particles flying through the sensor is detected). This signal is proportional to the amount of particles emitted by the engine. This technology provides a

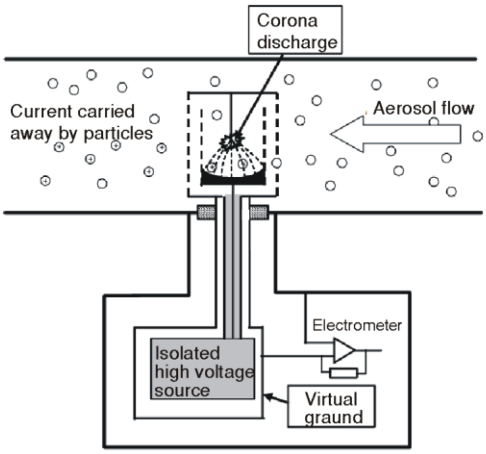

Figure 24. Dekati ETaPS

rapid response and extremely wide dynamic range. Response time is ~0.2 s and measured particle mass concentrations are from 0.01 to 100 mg/m^3. Due to a lack of dilution equipment, it is easily applicable for PM measurements.

Especially interesting is a new product of company Pegasor Ltd. from Finland [59]. They offer the ***Pegasor particle sensor*** (PPS), very compact and small instrument, which does not need special dilution (fig. 25) [60, 61]. It uses raw exhaust gas and can be installed near to the exhaust pipe in the vehicle or at the test bench. Actually, this instrument is a Faraday cup inside which particles are charged by corona ionizing charger and are pumped by an ejector dilutor built in the sensor's construction. At the entrance of sensor active part, fresh clean air is ionized by ~2 kV high voltage discharge at platinum corona needle. Positive ions are pushed through ejector nozzle, which generates under pressure for the suction of sample gas with particles. The ejector pump creates a constant sample flow through the sensor, which is not affected with flow variations inside exhaust pipe. After the ejector, turbulent mixing ensures good connections between ions and particles and deposit of certain quantity of ion on particle surface. Positive free ions (not deposited on particles) are removed from sample flow by positive trap voltage from central electrode. The generated electrical field pushes free ions towards the wall of sensor's body where they are collected and only ionized particles leave the sensor (fig. 25) [62]. An electrometer measures the difference between charge before (sample in) and after (sample out) faraday cup. This difference is proportional to the quantity particles (total mass or number) in exhaust sample. The sensor working principle is based on already mentioned ECT, *i. e.* the loss of current is proportional to the particle concentration [63].

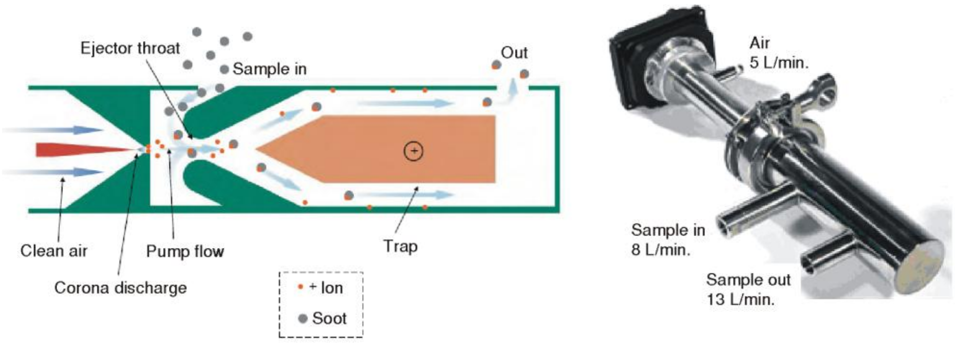

Figure 25. The schematic (left) and picture (right) of Pegasor particle sensor

The advantages of PPS are no need for dilution and real time measurement in continuous operation with high resolution (1 Hz) and response time of 0.3 s. In addition, sensor reading is independent of measured gas conditions (pressure fluctuations, flow velocity, operating temperature). The instrument can be installed permanently in the tailpipe, as well as upstream or downstream DPF. Measured concentration of particles varies from 0.01 to 250 mg/m^3 (fine and ultra fine particles). Special maintenance is not necessary

Conclusions

Having in mind all above mentioned, next conclusions can be drawn.

- Extremely low emission levels of modern diesel engines (EURO 5 and later) applying new technologies and aftertreatment have reached detection limits of current gravimetric measurement methods of particle mass emission. Therefore, gravimetric mass measurement of particle emission has to be improved.

- The particles mass is not only interesting parameter. Particles size, distribution, number, composition and surface can be also very important.
- Particle characterization can be performed with the instruments used in aerosol technique, such as: condensation particle counter (CPC), diffusion charger (DC), electrical low pressure impactor (ELPI), differential mobility analyzer (DMA), scanning mobility particle sizer (SMPS), *etc*.
- After several years of studies, PMP/GRPE/ECE/UN group has proposed the method and certification protocol for measurement of particle number. The method includes several dilution systems, an evaporation tube for elimination of volatile particle and a counter for evaluation of solid particle number. However, the PMP does not include volatile particles, which seem to be important to health.
- Special problem is whether exhaust emission of particles measured in laboratory conditions correspond to "real-life" particles emission of in-service diesel vehicles. To study this problem, EU has started so-called portable emission measurement systems (PEMS) project.
- At the moment, PEMS is measuring particle emission in real driving conditions using several available portable systems to obtain the insight in the possibility of these system for reliable measurement of particle emission in "real-life" conditions.

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Acronyms

CFV – constant flow venturi
CPC – condensation particle counter
CQCM – carousel quartz crystal micro balance
CVS – constant volume sampling
DC – diffusion charger
DCE – diffusion charging electrometer
DCS – diffusion charge sensor
DEED – Dekati engine exhaust diluter
DMA – differential mobility analyzer
DPF – diesel particulate filter
EAD – electrical aerosol detector
ECT – escaping charge technology
EECPC – engine exhaust condensation particle counter
EEPS – engine exhaust particle sizer
EFM – exhaust flow meter
ELPI – electrical low pressure impactor
EMS – electrical mobility spectrometer
ET – evaporation tube
FO – flow orifice
FPS – fine particle sampler
GRPE – representatives on pollution and energy
HEPA – high-efficiency particulate air
ISC – in-service conformity
MPS – micro proportional sample system
MSS – micro soot sensor
OBS-TRPM – on-board transient response particle measurement
PA – photoacoustic
PCF – cyclone pre-classifier
PEMS – portable emission measurement system
PFSS – partial flow sampling system
PM – particulate mater
PMP – particle measurement program
PNC – particle number counter
PPMD – portable particulate mass measuring device

PPS – Pegasor particle sensor
PSP – particle sampling probe
PTT – particle transfer tube
QCM – quartz crystal micro balance
SMPS – scanning mobility particle sizer
TD – thermodesorber
VPR – volatile particle remover

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