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**NanoMet:
on-line characterization of nanoparticle size and composition**

NanoMet: On-line Characterization of Nanoparticle Size and Composition

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Abstract

NanoMet is a new technique for on-line characterization of nanoparticle size and composition and their diffusion behavior. NanoMet consists of a pocket size diluter with tunable dilution ratio, a sampling interface for high concentration measurements and two on-line sensors. Simultaneous operation of the two sensors yields both the active surface (corona discharge diffusion charging sensor, DC) and the active surface times material coefficient (photoelectric aerosol sensor, PAS). Division of the readings provides the material coefficient which turns out to be characteristic of the particle source. Thus, information on source and toxicity of the aerosol is obtained. Thanks to the diluter and the sensitivity of the sensors the measurable concentration range stretches from (vehicle) raw emissions to ambient air / occupational exposure measurements. A particle sizing unit with a diffusion battery and a centrifuge is under development.

NanoMet measures particles *in-situ*, i.e. as aerosol. The time resolution (down to 1 second) and the sensitivity of the instrument facilitates transient measurements in the whole concentration range. For diesel soot, excellent correlation of NanoMet readings and the concentration of elemental carbon (EC) was found. The instrument is available in various configurations from tailpipe measurement systems fitting in a large suitcase down to pocket size sensors for occupational exposure monitoring. It is robust enough to be operated under fairly rough field conditions. The design can be simplified to a point where no special skills in particle measurement are required any more to operate NanoMet. This paper reports results and experience with a unit from the first series of equipment and discusses applications and accessories beyond the present state.

Introduction

The results of the VERT project (Mayer *et al.*, 1999) have demonstrated that state-of-the-art particulate traps are very well suited to reduce particle emissions from diesel engines to toxicologically tolerable limits. Once in use, however, the filtration efficiency of such traps, as well as the total particle emissions of the vehicle have to be periodically assessed. In order to minimize interruptions of vehicle availability for the owner, efficiency tests are to be carried out “in the field” without the need to dismount the trap from the vehicle, and the duration of the test itself should be kept as short as possible. Furthermore, the test method must be sensitive both at high and low particle concentrations as are found upstream and downstream of the trap, respectively, and it should be easy to operate for the user.

One of the merits of the VERT project is that particle measurement was not restricted to the approved legal methods but extended to any available laboratory technique. From this large pool of methods, it was not the legal ones that fulfilled all above criteria, as they are either not accurate enough or may not be applied under field conditions. Instead, a combination of a diluter and at least two on-line sensors - devices which had originally been designed for laboratory use - turned out to be the most suitable choice.

This result led to the VERT follow-up project “NanoMet” (*Nanoparticle Measurement Technique*), the goal of which is to integrate the on-line sensors and diluter in a portable, easy-to-use instrument for trap survey in the field. This paper reviews the working principles of the system and reports results and experiences with a unit from the first series.

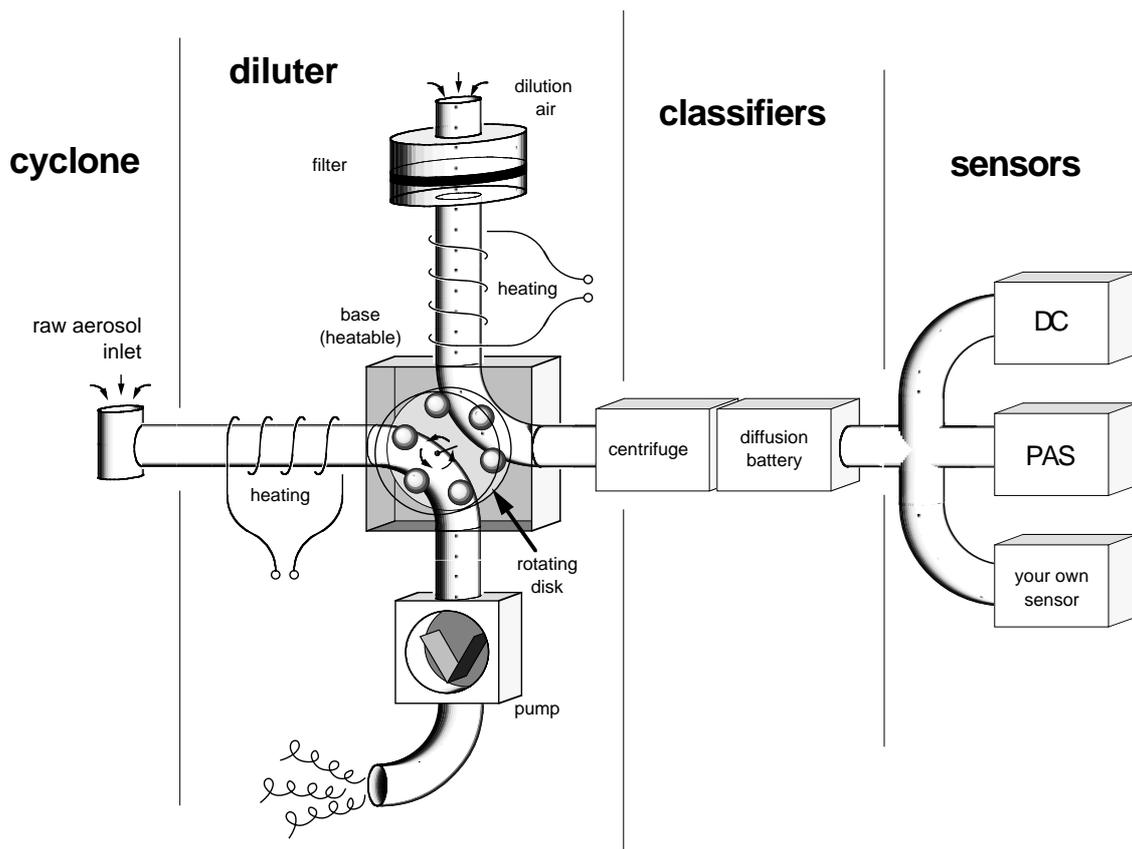


Figure 1: Schematic drawing of the NanoMet setup. Aerosol enters the cyclone and flows through the heated diluter and the classifiers to the sensors. Details are given in the text.

Setup and Working Principles

NanoMet

Fig. 1 shows a principal view of the NanoMet setup. A detailed description of it has been given by Matter (1998), which will be briefly reviewed here. NanoMet consists of a compact heatable diluter, a particle classifying unit with a centrifuge and a diffusion battery, and two on-line sensors. A cyclone at the sampling inlet keeps coarse particles out of the system. The time resolution of the system is determined by the rotation frequency of the diluter disk and the response time of the sensors. The current version achieves a lower limit of 1 second and for the near future a resolution of 0.1 seconds is planned. Reproducibility of measurements lies within a margin of a few per cent.

The NanoMet modules can be used as stand alone devices or in combination to best fit the user's needs. For example, ambient air measurements or occupational exposure assessment do not require the diluter due to low particle concentrations, but the use of both sensor types may be desirable. In another configuration with high particle concentrations, the user may wish to use the heatable diluter but complement or even replace the NanoMet sensors by his own technology.

Diluter

The diluter consists of a steel base through which two aerosol ducts are drilled, one for the undiluted aerosol as sampled from the source, the other for particle free air (Hueglin *et al.*, 1997). On one side of the base the channels are milled open. A rotating disk pressed against the polished surface of the base seals the orifices off against the environment. Small cavities in the back of the disk transport well-defined volumes from the raw aerosol orifice on the primary side into the particle free air on the secondary side from where the dilute aerosol flows to the sensors. The dilution ratio is determined by the rotation frequency of the disk and the flow rate on the dilute aerosol side. The orifices in the base are designed in such a way that primary and secondary channel are never in direct contact. Thus pressure fluctuations on one side cannot cause disturbance on the other.

Steel base and dilution air can be heated up to 150°C. This is useful when the raw aerosol is hot and dilution is to take place before the aerosol cools down to ambient temperature. In the measurement of tailpipe emissions this separation of dilution from cooling process prevents the condensation of volatiles, especially water and sulfuric acid. Selection of a lower heating temperature provokes condensation of some species while more volatile substances remain in gas phase. This feature can be used to distinguish between aerosol constituents that have different condensation temperatures.

Dilution is not needed if particle concentrations in the raw aerosol are low, such as downstream of a particle trap. In this case the diluter is easily bypassed with a valve. A separate unit holds the power supply, a pump for the raw aerosol, the controls for the diluter disk motor and heating. Supply unit and diluter head are interconnected by shock and heat protected cables and hoses. The most important technical specifications are listed in Table 1.

	diluter
dimensions of supply unit	258 x 148 x 312 mm 1/2-19" plug-in unit
dimensions of diluter head	360 x 180 x 90 mm
length of connecting hoses	up to 3 m
raw aerosol flow	1 l/min
dilution air flow	0.5 ... 5.0 l/min, determined by connected sensors
disk rotation frequency	0.15 ... 3.0 Hz
dilution ratio	1:30 ... 1:600 (disk with 10 cavities) 1:150 ... 1:3000 (disk with 2 cavities)
heating temperature	OFF / 80°C / 120°C / 150°C

Table 1: Selected properties of the diluter.

Classification

Three classifying elements are used in NanoMet. A cyclone with cut size 2.5 µm in the raw aerosol channel prevents coarse particles from entering the diluter. Behind the diluter a centrifuge removes particles larger than 200 nm. Its cutoff can be varied towards larger diameters by tuning the rotation frequency. The third classifier is a multi-stage diffusion battery located after the centrifuge with cutoff at 4 different mobility diameters. Size classi-

fication facilitates the measurement of size spectra; in combination with the DC sensor reading the number concentration per size class can be calculated. As the classifier modules are still under development, no measurement data is shown. Data presented in this report was measured without size classification.

In the particle centrifuge, an aerosol stream is exposed to the centrifugal field of a rotating drum forming a solid body vortex. In the flow field, a circular gap between an outer and an inner cylinder, the particles are forced to the outward. Thus regions of enriched and diluted particle concentrations will develop. Unlike in older designs reported in the literature, where the separated particles were collected on a foil cladding of the inner surface for later determination of the size distribution, the classification serves to produce an aerosol flow fraction reduced of particles larger than the size class of interest, which is fed to the measuring devices downstream.

The centrifuge has two adjustable parameters: Speed (centrifugal force) and throughput (residual time). So it is a freely tunable particle classifier. When operated, it also acts as a ventilator, this is why the cut size is not coupled with pressure drop. Though for the application in NanoMet the centrifuge's significance will be that of an adjustable preseparator, it can be also used for classifying particles according to their aerodynamic diameter down to 50 nm or below. After having tested a first design which shows the feasibility but has grade efficiency curves of unsatisfactory steepness, a new outlay is now under investigation.

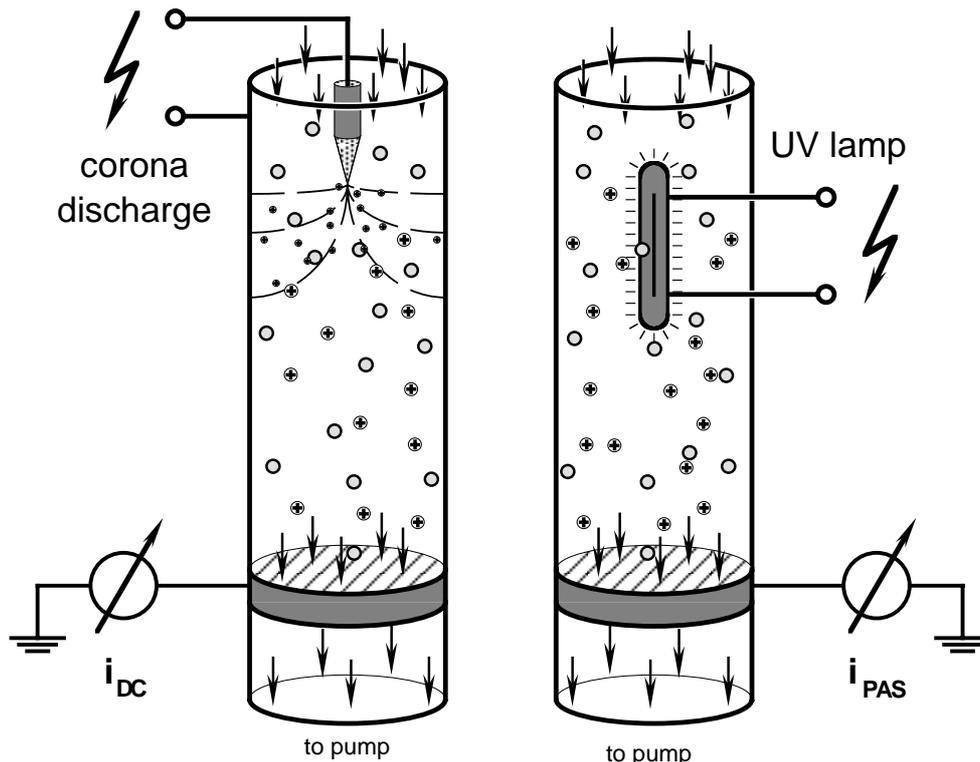
For the size classification of particles according to their mobility diameter, a diffusion battery is applied. It is of tube type with layers of wire gauze. The adjustable parameters are the number of gauze layers and the volume flow. The present design comprises a series of layer packages with exits in between through which the reduced gas stream is fed to the connected sensors. Cut sizes of 15, 35, 75 and 190 nm could be realized. Even though the penetration curves are rather flat, they can be clearly distinguished for the given diameters.

Sensors

NanoMet comes with two on-line sensors where particles are electrically charged and subsequently precipitated on a filter (Fig. 2). The electric current flowing from the filter is amplified and measured. The sensors differ in the charging principle - in the diffusion charger (referred to as DC) positive ions from a corona discharge diffuse onto the particles. Therefore, the filter current is proportional to the total active surface of the aerosol, ΣS_{active} . A detailed derivation of this concept is given in the next section. In the photoelectric aerosol sensor (referred to as PAS) aerosol particles are illuminated by ultraviolet light and photoelectrically charged. The electrons are then quickly removed from the gas by an electric filter. As photo emission involves absorption of a photon by the particle bulk material and emission of an electron through the particle surface, the resulting charge on the particles is proportional to the active surface, ΣS_{active} , and a material coefficient, ϕ . This material coefficient is especially large for solid particles from fossil fuel combustion.

Simultaneous operation of the two NanoMet sensors yields both the active surface (DC sensor) and the active surface times material coefficient (PAS). In the experiment, the respective aerosol is dispersed homogeneously in a small office room. Samples are drawn from the room air through a common pipe for the two sensors to ensure that local fluctuations in particle concentration does not cause divergence of the signals. Division of the readings provides the material coefficient which turns out to be characteristic of the particle source. Fig. 3 shows a plot of PAS vs. DC signal for particles from various sources and the respective material coefficients. The readings show linear correlation over a large concentration range but with individual slope for each material. A very high photoelectric activity is observed for soot from candles and wood fire while cigarette smoke and iron oxide exhibit little such activity. Droplets of water or condensed organic materials do not undergo photo emission at all - their material coefficient is zero.

The measurable particle size range of both sensors is in principle unlimited towards the lower end. At the upper end, the PAS cuts at approximately 1 micron because the emitted electrons will be drawn back to the surface of the charged particle. Only if the particle is smaller than the mean free path of the electron in the gas (~ 1 micron)



$$i_{DC} \propto \sum S_{active}$$

$$i_{PAS} \propto \sum S_{active} \cdot \phi$$

i_{DC}, i_{PAS}	filter current of DC sensor / PAS
S_{active}	active surface of a single particle
ϕ	material coefficient of the aerosol
Σ	sum over all particles

Figure 2: Working principles of the diffusion charging sensor (DC, left) and the photoelectric aerosol sensor (PAS, right). The measured current is proportional to the total active surface of the aerosol. The PAS signal further depends on a material coefficient.

the electrons will fail to find the particle and be lost in the gas. Thus only submicron particles remain charged and contribute to the signal. For diffusion charging with the DC sensor no upper size limit exists, but the cyclone, centrifuge and diffusion battery keep coarse grains out of the system.

Portable versions of the two sensors are available. These battery supplied devices can be operated for up to 6 hours while saving data in the internal logger. Being small and lightweight they may be carried in a back pack or camera pocket for personal monitoring.

The most important properties of all sensors are listed in Table 2.

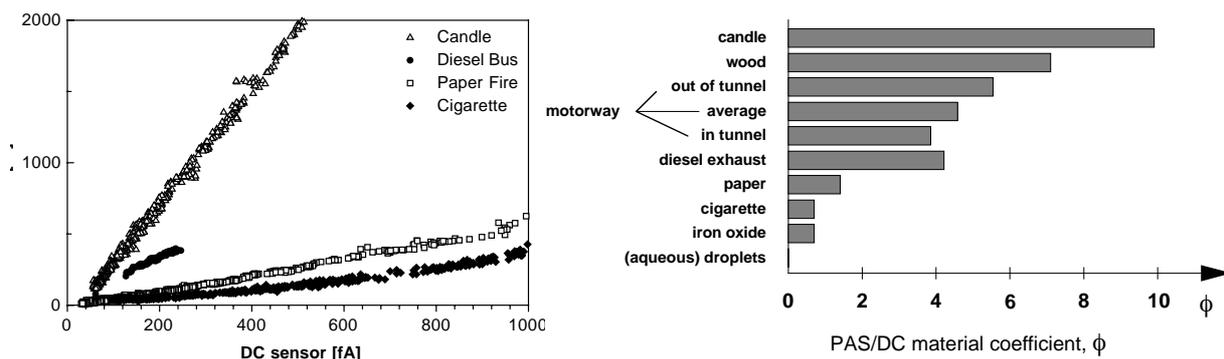


Figure 3: Simultaneous measurement of aerosols from different sources using PAS and DC sensor. The signals show linear correlation but with different slopes for each material. Selected material coefficients are plotted in the right graph.

Theory and Calibration

Active Surface

In the DC sensor, ions from the corona discharge diffuse between the neutral gas molecules and eventually reach the surface of an aerosol particle. Thus the DC measures the integral attachment cross section of ions colliding with the aerosol particles. As a sticking coefficient of one can be assumed for ions, this equals the collision cross section or collision frequency. If the ion concentration is kept low enough to avoid multiple charging, the collision frequencies of ions and neutral atoms are alike, otherwise Coulomb repulsion will decrease the ion attachment rate. Furthermore, the aerosol particles have to be larger than about 10 nm because only in this case the image force can be neglected. For smaller particles the image force will increase the attachment probability (Filipov, 1993). This implies a slight overrepresentation of ultrafine particles in the DC signal. However, this is negligible in most applications..

	DC sensor	DC - portable	PAS	PAS - portable
dimensions	258 x 148 x 312 mm 1/2-19" plug-in unit	70 x 130 x 170 mm 1.5 kg	258 x 148 x 312 mm 1/2-19" plug-in unit	70 x 130 x 170 mm 1.5 kg
battery operation	-	up to 6 hrs	-	up to 6 hrs
physical property being measured	active surface, independent of material		active surface, material specific (carbonaceous soot)	
measurement range (filter current)	0 - 1000 fA	0 - 2000 fA	0 - 5000 fA	0 - 2000 fA

Table 2: Selected properties of the NanoMet sensors.

	DC sensor	DC - portable	PAS	PAS - portable
resolution, corresponding to -	1 fA = 10^{-15} A $1 \mu\text{m}^2/\text{cm}^3$	3 fA = $3 \cdot 10^{-15}$ A $3 \mu\text{m}^2/\text{cm}^3$	1 fA = 10^{-15} A $0.125 \mu\text{g}/\text{m}^3$ EC (only diesel exhaust)	3 fA = $3 \cdot 10^{-15}$ A $0.375 \mu\text{g}/\text{m}^3$ EC (only diesel exhaust)
particle size range	1 nm ... 2.5 μm (integral)		1 nm ... 1 μm (integral)	
aerosol flow	1.5 l/min	1 l/min	2 l/min	1 l/min
time resolution	< 1 sec	~ 10 sec	~ 1 sec	~ 10 sec

Table 2: Selected properties of the NanoMet sensors.

If these conditions are fulfilled, the DC sensor measures the integral collision cross section or attachment cross section, respectively. This is an important quantity by itself, as it determines adsorption kinetics and thereby has significant influence on chemical reactions between particles and the surrounding gas phase or gas phase reactions where the particles serve as catalyst. It also determines particle growth by attachment of material from the gas phase. One could say it is the fraction of geometric surface which is directly accessible from outside. For this reason the term ‘active surface’, S_{active} , is used. A related designation, ‘Fuchs-Surface’, has been introduced in connection with the Epiphaniometer (Pandis *et al.*, 1991).

Mobility and Active Surface

Another important physical property of aerosol particles is their mobility, b , which determines the velocity v a particle obtains as response to the action of an external drag force: $v = b \cdot F$. The mobility is measured with a differential mobility analyzer (DMA) where it is inversely proportional to the applied voltage. Furthermore, b is related to the diffusion constant, $D = b \cdot kT$, where k is Boltzmann’s constant and T the temperature. D , in turn, determines the probability that the particle will diffuse to the walls of a vessel containing the aerosol.

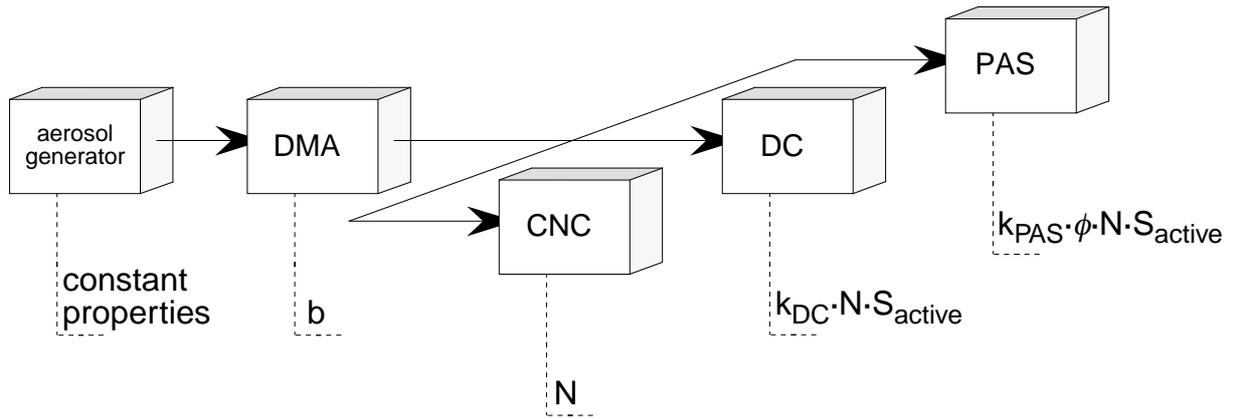
The two concepts of microscopic active surface, S_{active} , and macroscopic mobility, b , are connected by an astonishingly simple relation, $S_{active} \cdot b = const$, which holds over the whole diameter range within an uncertainty of a few percent independently of particle size, shape or material (Keller *et al.*, 1999). This is plausible as the drag force, F , is also due to collisions with carrier gas molecules. The collisions determine the momentum transfer and thereby also the drag force.

Thus, inverse proportionality of the DC signal with the diffusion coefficient is established via active surface and mobility. The reading of the DC sensor may then be considered a direct measure of the amount of particles deposited in a tubing system such as the bronchial epithelium of the human lungs.

Calibration Reference Instruments

Calibration of the DC sensor is carried out using an aerosol generator and acknowledged reference instrumentation for mobility analysis and particle counting such as the hardware components of a SMPS system (Scanning Mobility Particle Sizer, TSI Inc., St.Paul MN, USA).

The calibration setup is shown in Fig. 4. Test aerosol is fed into a differential mobility analyzer (DMA) where a mobility class is selected. This monodisperse aerosol flow is split in two and guided to a condensation nucleus counter (CNC) and the DC sensor. In this setup, the test aerosol may consist of arbitrary material; particle number concentration and median diameter should be somewhat constant. At the DMA exit, the particle mobility, b , is uniquely determined by the DMA parameters voltage, air flows, and geometry, without need to calibrate the



$$S_{\text{active}} \cdot b = \text{const}$$

Figure 4: Schematic drawing showing the calibration setup and properties measured by each instrument, and the calibration equation.

instrument. Particle number concentration, N , is measured in the CNC, and the DC sensor captures the total active surface of the monodisperse aerosol, $S_{\text{active}} \cdot N$. The only parameter remaining to be tuned is the amplification factor in the DC sensor, k_{DC} . This concludes the calibration process.

Calibration Aerosol

The PAS is calibrated in the same setup. As the reading of the PAS differs from that of the DC sensor by a multiplicative material coefficient, ϕ , a special test aerosol must be used. While there are no strict requirements for the test aerosol for calibration of the DC sensor, the PAS needs particles of certain chemical composition with constant, reproducible, and high ϕ values. The preferred test aerosol would be carbonaceous soot particles from the combustion of some hydrocarbon fuel. Using such a test aerosol the PAS could be calibrated against the DC sensor by simply tuning the PAS/DC ratio, ϕ , to the characteristic value of the aerosol. However, in the past no commercial aerosol generator of sufficient stability and reproducibility was available.

Recently a new device developed at the Swiss Federal Office of Metrology (Eidgenössisches Amt für Messwesen, EAM) has been introduced that fulfills these conditions (Jing, 1999). Combustion aerosol from a diffusion flame is quenched and diluted in inert gas and may then be used for calibration or other laboratory purposes. Particle sizes in the range 30–200 nm and concentrations around 10^6 cm^{-3} can be realized. Size distributions and material coefficient were shown to be stable and reproducible within a few percent.

Aerosol Characterization

Instrument Demands

The term *aerosol* designating particles in gas suspension is a very general expression for a highly complex system. Aerosols are far from being stable. The particles diffuse through the gas on random paths, eventually colliding with each other. As surface forces are very strong, the particles will stick and a single, larger particle emerges

from the collision. In the aerosol ensemble, coagulation and agglomeration make the particle concentration decrease as time passes while the average diameter increases. At the same time, particles interact with the surrounding gas molecules in chemical reactions, vapors condense to form droplets or volatile particles evaporate.

Moreover, ambient aerosol particles are individuals - no two of them are alike. Even so well-defined an aerosol source as a diesel engine releases to the environment a large variety of particles in the submicron size range. A major part of mass and number is concentrated in solid carbonaceous soot particles. Ashes and minerals from engine wear or additives make up for the rest of the solid granules. But this is only one half of the nanoworld. Water and inorganic acids, mostly sulfuric acid, condense to droplets which often exceed the solid particles by far both in number and in mass.

This inhomogeneous mixture of solid soot, salts and volatiles fills the air that we breathe. It is obvious that water droplets in the human lung will not yield the same effect as carbonaceous soot or minerals. If, on the other hand, particle emissions are to be efficiently reduced the source must be identified. In the diesel engine carbonaceous soot is a result of the engine's working principle whereas sulfuric acid originates from sulfur in the fuel.

A measurement technique that characterizes ambient or emission aerosol must be able to distinguish particles with different physical and chemical properties, and it should be fast enough to allow for changes in aerosol composition during sampling. NanoMet characterizes particles in two steps. First, droplets are separated from solid particles using the heatable diluter. Then the material coefficient, ϕ , of the dilute mixture is compared to known or calibrated values. The following examples illustrate our method.

Distinction Solid - Liquid

The NanoMet approach to treat droplets as different species aims at preventing them from ever condensing at all. This is accomplished by heating the diluter head and dilution air to 150°C, which sets the process of aerosol dilution prior to cooling of the mixture to room temperature. Thereby the order of the two processes - dilution and cooling - is reversed, as opposed to the dilution tunnel where conditions are most favorable for the formation of droplets. Furthermore, two lower heating temperatures can be chosen (120°C, 80°C) at which some of the less volatile compounds may condense while others remain in gas phase. This allows one to further characterize the volatile components of the aerosol, e.g., weakly acid water vapor will not condense at 120°C while vapor of more concentrated sulfuric acid will.

The principle is illustrated in Fig. 5. As temperature in the heated diluter head is increased, the reading of the "overall surface" DC sensor decreases. The PAS detects an almost constant concentration of solid carbonaceous soot particles at any temperature.

Distinction Soot - Mineral

An application of the material coefficient measurement of NanoMet is shown in Fig. 6. Emissions of a diesel engine operated with two types of fuel were characterized using SMPS and NanoMet. With standard fuel the SMPS spectrum shows the usual log-normal distribution peaking at 70 nm. The NanoMet reading is equal for both sensors, which can be interpreted as "all particles" (DC) are "soot particles" (PAS). In the second case the fuel was doped with some organo-metallic compound. The effects of this additive on the emission spectrum measured with a scanning mobility particle sizer (SMPS) are twofold: (a) The number of soot particles is reduced by half an order of magnitude. This result is reflected in a reduction of the PAS signal to a third of its previous value. (b) The second effect is that the additive generates new small particles in high numbers which are suspected not to be soot particles. NanoMet confirms this suggestion by an almost tripled DC reading. Thus the relevant information - soot reduction while total particle emissions are increased - is obtained in a very straightforward manner without the need for additional measurements or chemical analysis.

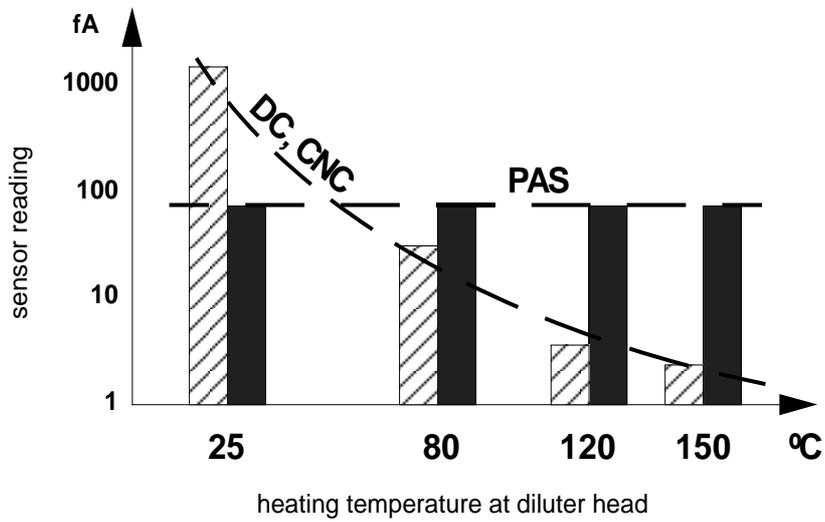


Figure 5: Droplets are detected by the DC sensor (or a Condensation Nucleus Counter, CNC) but not by the PAS. As the diluter head is heated to higher temperature less volatiles condense. The DC signal decreases while the PAS displays a constant value because solid soot particles are not affected by heating.

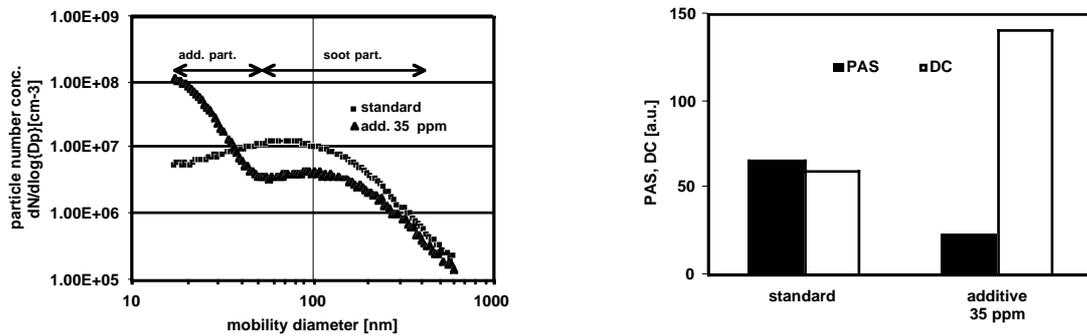


Figure 6: Emission measurements with scanning mobility particle sizer (SMPS, left) and NanoMet (right) at a diesel engine operated with standard fuel and cerium doped fuel. Due to the additive the number of soot particles is decreased while the total particle number increases (SMPS spectra). This result is also reflected in the PAS and DC signals.

Field Use and Correlations

Owing to the high sensitivity of the PAS and DC sensor, combined with the diluter, the whole concentration range between emission and ambient air / occupational exposure measurement can be covered. With NanoMet one has an instrument at hand which solves the unsatisfactory situation that the same particles are measured and legally limited by different, often not even correlating methods, only depending on the present legal situation. Numerous ambient air and emission measurements have been carried out during the last years where sensors and diluter

	engine emissions		engine emissions and ambient air	ambient air
source	heavy duty diesel engines (test bench)	diesel passenger car (roller dynamometer)	generator diesel engine; road traffic	road traffic
reference quantity	non-soluble organic fraction	number concentration of 100 nm particles	induction factor of mutations in bacteria	non-soluble organic fraction
reference instrument	-	CNC	bacteria: <i>salmonella typhimurium</i>	aethalometer
concentration range	0...66 mg EC / m ³	0...600 µg EC / m ³	0...0.06 µg PAH / m ³	0...18 µg EC / m ³
correlation coefficient	0.97	0.97	0.82	0.90
author	FVV report, 1997	Matter <i>et al.</i> , 1999a	Wasserkort <i>et al.</i> , 1998	BRISKA / Gysin, 1999

Table 3: Correlations of the PAS signal in various applications.

worked under field conditions.

Excellent correlations of the PAS signal have been found for elemental carbon (EC) concentrations in ambient air (BRISKA/Gysin, 1999) and for the non-soluble organic fraction in the emissions of heavy duty diesel engines (FVV report, 1997). Due to its good time resolution, the PAS can be used for transient measurements such as driving cycles (Fig. 7). Being an indicator of genotoxicity in biological systems (Wasserkort *et al.*, 1998) the PAS is well suited for personal exposure monitoring. The most important correlations of the PAS signal are listed in Table 3.

Prototypes of the centrifuge and diffusion battery have yielded encouraging test results and will soon be integrated as new NanoMet modules.

Hardware - The NanoMet Family

Broad range applications require variety in instrumentation. In the NanoMet system, a number of accessories are grouped around a few core modules. Their combination is determined by the user's needs. Examples are given in Table 4.

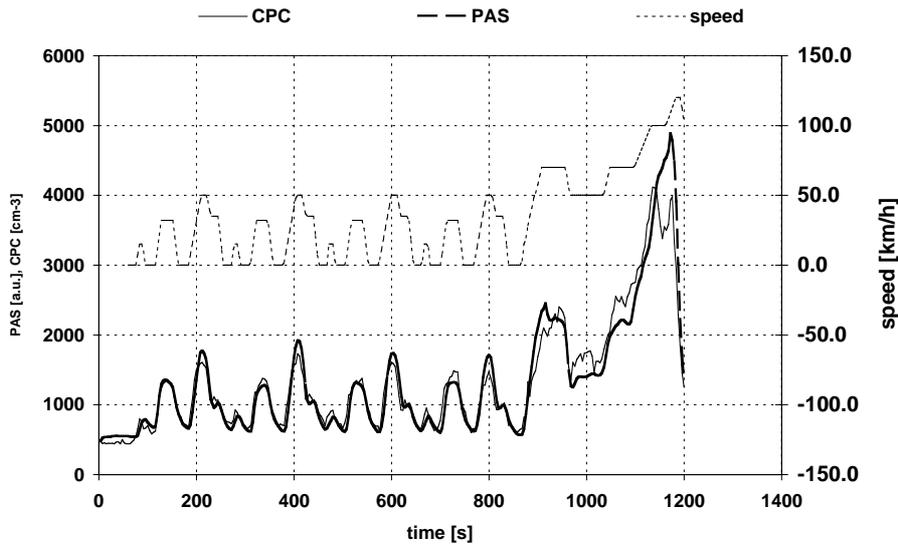


Figure 7: Particle emission measurement during a New European Driving Cycle (NEUDC). The PAS signal correlates well with the number concentration of 100 nm particles recorded by a CNC.

application	DC sensor	PAS	diluter	sampling interface	classifiers	aerosol generator	diluter in 19" rack	PAS portable	DC portable
tailpipe emissions; trap efficiency test (field measurements)	(X)	X	X	X					
characterization of tailpipe emissions	X	X	X	X	X				
process control in large scale aerosol production	X	X	X		X				
ambient air survey	X	X			X				
production of defined aerosol (for calibration or lab use)	(X)	(X)				X	X		
occupational exposure; personal monitoring								X	X

Table 4: Applications in which the use of NanoMet has been tested, and the corresponding set of modules.

Summary

The newly developed NanoMet technology that emerged from the results of the VERT project has passed its first field tests. According to the initial goals of the project, NanoMet measures particles *in-situ*, i.e. as aerosol. The time resolution (1 second) and the sensitivity of the instrument facilitate transient measurements in a concentration range stretching from ambient air up to vehicle emissions. NanoMet distinguishes particles of different chem-

ical composition and characterizes their diffusion behavior. Thus, information on source and toxicity of the aerosol is obtained. The instrument fits in a large suitcase and is robust enough to be operated under fairly rough field conditions. The design can be simplified to a point where no special skills in particle measurement are required any more to operate NanoMet.

Acknowledgments

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