



# *2013 Cambridge Particle Meeting*

## *Program abstracts*

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Chairmen:

**Adam Boies** and **Jacob Swanson** (University of Cambridge)

**Jon Symonds** and **Chris Nickolaus** (Cambustion)



## In-flame soot diagnostics and particle emissions of full-scale aero engine fuel injectors

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Characterization and reduction of aircraft particulate emissions have received considerable emphasis in recent years due to growing concerns related to nanoparticle emissions and forthcoming regulations. In aero engines, the most effective point of control for emissions is recognized as the fuel injector and combustor. Therefore investigations at these stages are essential for building the capability to reduce emissions through improved combustion technologies.

The proposed talk provides an overview of soot research at the Cambridge Intermediate Pressure Combustion Facility, which has been underway since 2009, supporting gas turbine technology development programmes of Rolls-Royce plc. The facility enables operation at pressures up to 10 bar, with air preheat temperatures up to 600°C and continuous preheated air flow capacity up to 0.9 kg/s. The capability to accommodate full-size fuel injectors and extensive optical access allow detailed investigation of the combustion zone at realistic conditions. Both lean burn and rich burn technologies have been tested throughout the study on various prototype fuel injectors operated on kerosene.

A suite of in-situ optical diagnostic techniques have been implemented to map out soot distribution within the combustor and its relation to the earlier chain of events from fuel spray, droplet atomization, evaporation and reaction. Planar laser induced incandescence (LII) and laser extinction were utilized for in-situ soot measurements. Mie scattering, planar laser induced fluorescence (LIF) and OH\* and CH\* chemiluminescence were used for visualization of liquid and vapour distribution and reaction zones. Effects of operating conditions such as inlet temperature, pressure, mass flow rate, air-fuel ratio and cooling air were investigated.

In-situ measurements were compared with ex-situ measurements sampled from the combustor exit. Gaseous emissions of CO<sub>2</sub>, CO, NO, NO<sub>x</sub> and unburnt hydrocarbons were measured. Total particle number concentration and size distribution were measured using a Scanning Mobility Particle Sizer (SMPS). A Hartridge smokemeter was also used to provide comparison against more conventional smoke measurement tools of the aerospace industry.

The results improve understanding of the soot processes starting from the fuel injector through to particle emissions at practically relevant conditions.

## Fabricating solid-state gas sensors by aerosol-based techniques

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Gas sensors are of crucial importance for ensuring the safe operation of a wide number of energy conversion processes. Energy production from fossil fuels, for example, results into emissions of gaseous species (e.g., CO, SO<sub>2</sub>, NO<sub>x</sub>, etc.) that are threatening to human health and the environment, and thus need to be continuously monitored. Although the perspective of replacing fossil fuels with H<sub>2</sub> can drastically reduce the emission of many of these unwanted species in the future, its safe handling during production and storage requires sensitive and robust hydrogen sensors. Despite the great efforts in fabricating such sensors, most of them have the disadvantage of being technologically complex and expensive. Methods for producing simple and cost-effective sensors are therefore highly needed.

In this presentation I will describe a number of aerosol-based techniques for fabricating nanomaterials that can be employed in gas sensors. Aerosol-based techniques offer great opportunities for synthesizing nanoparticles of well-defined composition, size, and morphology. In addition they offer tools for producing versatile nanoporous structures with granular and/or dendritic morphologies, thereby allowing good control over their surface-to-volume ratio. By further annealing the resulting nanomaterials one can also control their crystal structure, which in turn affects their behaviour upon adsorption/desorption of gaseous species on their surface. The feasibility of some of these techniques will be demonstrated by showing how WO<sub>3</sub>- and Pd-based nanostructured materials can be fabricated and employed in NO<sub>x</sub> and H<sub>2</sub> gas sensors, respectively.

## Sooting tendency of paraffin surrogates of diesel and gasoline in diffusion flames

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The influence of the chemical structure in the sooting characteristics of surrogates for the paraffinic fraction of gasoline and diesel fuel is studied experimentally. The experiment involves the combustion of the paraffin in a smoke point lamp (ASTM D1322). Differential mobility spectrometry is used to measure the particle size distribution (PSD) at different flame heights. The wick-fed laminar diffusion flame is sampled at the tip; the flame height is modified systematically from small heights to large heights (in some cases beyond the smoke point). Normal, iso and cyclo paraffins PSDs evolve in a similar way with flame height. At very low flame heights the PSD is unimodal, but rapidly evolves to a multi-modal one. The total number of particles decreases with flame height and approaches constant values for all considered fuels. As flame height increases so does the mean soot particle diameter until a height where a maximum is achieved and sustained. As the number of carbon atoms increases in the molecule the final mean soot particle diameter decreases. Cyclic and iso-paraffins produce soot particles with larger mean sizes compared to normal paraffins of the same carbon number.

[1] ASTM. Standard test method for smoke point of kerosene and aviation turbine fuel. ASTM Standard D1322-08.

## Hot condensation particle counter

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This work is concerned with the development of a novel high-temperature CPC (HT-CPC) as an alternative way of measuring solid particles. The aim is to design and build the HT-CPC that is by design insensitive to volatile particles, by operating at such a high temperature that volatile material is evaporated and cannot re-condensing. The HT-CPC will eliminate the need for cooling and problems associated with cooling. Moreover, it could replace the complex PMP system by a single device, and perhaps also of significance, be able to measure solid particles down to very small sizes if required.

Essential for the design process, a theoretical model of heat and mass transfer in the HT-CPC was constructed. The model allowed key parameters such as Kelvin-equivalent counting efficiency, volatile removal efficiency and homogeneous nucleation to be predicted. The simulations suggested that the HT-CPC will be able to grow and detect solid nanoparticles. A prototype was then constructed based on the model simulations.

One crucial design criterion was selection of high-temperature working fluids. The short listed candidates including organic oils (DEHS), perfluorocarbons (e.g. Fomblin fluids), silicone fluids (Dow Corning 704, 705 or equivalents) and poly-phenylethers (e.g. Santovac 5) were tested experimentally.

The experimental set-up comprised an aerosol generator, a differential mobility Analyzer (DMA), a TSI 3775 butanol CPC and the HT-CPC. Test aerosols used in the experiments were NaCl, lab ambient particles, combustion generated particles (Combustion DPG) and tetracontane particles.

Without sample pre-heating or dilution, the HT-CPC using DEHS, DC704 and Lesker705 was able to grow NaCl solid particles as predicted by the model. It successfully removed tetracontane particles of electrical mobility diameter range 7nm to 310nm at concentration higher than  $10^4$  particles/cc with about 99% efficiency. Unfortunately, DEHS decomposed at high temperatures with time making it unsuitable. Also perfluorocarbons and a poly-phenylether were found to be unable to grow or detect NaCl particles. This may have been due to the failure of the vapours to “wet” the particles’ surface, though this has not been established.

The HT-CPC was successfully built and tested. Using 704 and 705 silicone fluids, the HT-CPC could detect NaCl and soot particles as predicted by theory. It was able to remove tetracontane volatile particles up to 99% efficiency. The HT-CPC allows solid particles to be measured using just a single device.

## Gas-phase synthesis of magnetic/plasmonic nanoparticles for cancer theranostics

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Multicomponent, multi-layer nanoparticles have potential applications in cancer theranostics, where the different nanoparticle chemical components and layers enable imaging, heating, drug delivery and targeting. Key challenges for manufacturing such nanoparticles include control of the dimensions, morphology, chemical composition and functional properties of each layer, avoidance of impurity residues, stability in aqueous suspension, and scalability to commercially relevant production rates.

We have developed an approach to producing multilayer magnetic/plasmonic nanoparticles that is based on sequential gas-phase processing. Here we report use of this approach for production of nanoparticles consisting of a superparamagnetic iron oxide core that is encapsulated in successive layers of silica, gold and polyethylene glycol (PEG). The PEGylated nanoparticles are then dispersed into stable aqueous suspension. The iron oxide cores are produced in a plasma, and are then transported in a carrier gas through several chambers that sequentially grow layers of silica, gold and PEG. Processes utilized include UV photoinduced chemical vapor deposition for the silica, hot-wire generation of sub-5-nm gold nanoparticles, particle scavenging for attachment of gold onto silica, and PEGylation by attaching a bifunctional thiol to the gold and then inserting ethylene oxide into the hydroxyl end of the thiol. The resulting particles are readily dispersible in water, and can be further functionalized for active targeting and/or drug delivery. Total diameters of the multicomponent nanoparticles are typically in the range 20-50 nm, which is suitable for transport and deposition in tumors.

Several methods are used for online characterization of nanoparticles and of individual layers as they are produced, including scanning mobility particle sizing, tandem differential mobility analysis and in-situ Fourier transform infrared (FTIR) spectroscopy. Particles are also collected onto transmission electron microscopy (TEM) grids for offline characterization by TEM and related high-resolution chemical characterization. Bulk powder samples are collected on filters for characterization by X-ray diffraction, FTIR, magnetic property measurement instruments, and optical absorption and fluorescence.

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## Wall flow filter for particulate emission reduction of petrol engines

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Today the paramount driver in the automotive industry besides comfort and safety is the reduction of emissions and fuel consumption especially with regards to the stringent Euro 6 legislation and CO<sub>2</sub> targets for the year 2020.

While Gasoline Direct Injection (GDI) in combination with turbocharging seems to be an effective technology to increase the quality of mixture formation and thus thermal efficiency it has the side effect of increased particle formation during combustion compared to conventional petrol engines with port fuel injectors.

In 2012 the European Commission has agreed to implement a particle number limit that is equal to that of diesel engines (6E11 /km) and will come into force as part of Euro 6c in 2017.

For this purpose it has become necessary to investigate the feasibility of reducing gasoline particles by means of a Gasoline Particulate Filter (GPF). There are two main concepts of this technology:

1. Non-catalysed GPF
2. Catalysed GPF for integrated TWC function

This presentation focuses on the second concept, which is evaluated on different Euro 5 vehicles and - with respect to the latest proceedings for new test cycles - under different transient driving conditions.

The key for our development was to provide a suitable material for balancing filtration efficiency and induced pressure drop. The results show that NGK's selected cordierite material for GPF can undercut the future particle number limit of 6E11 /km during all selected driving cycles and therefore has potential to sustain high filtration efficiency even during real driving emissions (RDE) perhaps as part of certification for future post Euro 6 legislation.

## Issues associated with measuring nothing

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Increasing stringent mass emission standards are making it very difficult to make the measurements with any certainty. The current Euro 6 passenger car limit is 4.5 mg/km which can still be measured with reasonable confidence but planned California / US standards of 1.9 mg/km for 2017 and especially 0.6 mg/km for 2025 will challenge current gravimetric measurement technology. The 2025 mass emission standard is roughly equivalent to the current Euro 6 number standard of  $6 \times 10^{11}$  particle/km and measurements at this level are now regularly made without great difficulty. The problem is not making particle emission measurements but rather making gravimetric filter based particulate mass emission measurements at this level. Issues associated with these measurements will be discussed along with possible solutions.



## Flow of nanoparticles in and around road vehicles

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The air around us in typical outdoor urban environments contains 10's of thousand tiny sized particles. Consequently the city dwellers generally end up with a respiratory deposition of ~10-80 billion of particles each hour while walking or travelling along the busy roadsides. Over 99% of these particles, by number, are generally below 300 nm in size range. A dominant fraction of them are contributed by road vehicles in polluted urban environments. Due to competing influences of various transformation processes as well as varying strength of emission sources and dispersion conditions, nanoparticle concentrations may vary substantially in local (i.e. street canyons,  $\sim 10^4\text{--}10^6\text{ cm}^{-3}$ ) and micro-transport (vehicle wake and inside car cabins,  $\sim 10^3\text{--}10^7\text{ cm}^{-3}$ ) environments, and so is the case of the extent of public exposure. These remain currently unregulated through ambient air quality standards across the world. Recent Euro 5 and Euro 6 vehicle emission standards have included first ever limits for nanoparticles emissions at source on a number basis. Similar initiatives are needed to control public exposure to airborne nanoparticles. This talk will present some of the results of our recent EPSRC projects focusing on the concentrations and dynamics of nanoparticles in and around the road vehicles through experimental and physical modelling techniques, besides highlighting the challenges in their dispersion modelling at various spatial scales.

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## **Measurement of aircraft non-volatile PM emissions using aerospace recommended practice compliant systems during the A-PRIDE 4 campaign**

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The Society of Automotive Engineers (SAE) Aircraft Exhaust Emissions Measurement Committee (E-31) has defined a draft Aerospace Recommended Practice (ARP) sampling system for the measurement of non-volatile PM from gas turbine engines. The system is designed to operate in parallel with existing International Civil Aviation Organization (ICAO) Annex 16 compliant combustion gas sampling systems used for emissions certification from gas turbine engines captured by conventional (Annex 16) gas sampling rakes. Prototype versions of the draft ARP compliant sampling system – Missouri University of Science and Technology (MST) and the Swiss FOCA/EMPA systems – have been developed and demonstrated during testing opportunities as part of the Aviation Particle Regulatory Instrumentation Demonstration Experiment 4 (A-PRIDE 4) campaign at the SR Technics facilities in Zurich, Switzerland in November 2012. The primary objective of A-PRIDE 4 was the performance evaluation and inter-comparison of two draft ARP compliant systems. The MST and FOCA/EMPA systems were inter-compared during a series of dedicated engine tests using a CFM56-5B engine, and piggy back testing using CFM56-7B and PW4168 engines at a range of engine power conditions. Overall, these two draft ARP compliant systems were found to be within 5% of each other, in terms of non-volatile PM number and mass inter-comparison.

## Particulate emissions from seven petrol-engined light-duty vehicles taken from the European fleet

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A rolling test programme has been conducted by AECC over the last five years to provide data on the emissions of petrol-engined light-duty vehicles. The work has included the measurement of Particulate Mass and PM Number emissions using the light-duty PMP procedures developed under UNECE – GRPE.

The vehicle range covered two stoichiometric port fuel injection vehicles, three stoichiometric direct injection petrol vehicles, and two lean-burn direct injection petrol vehicles. The emissions levels of the vehicles covered the technologies used to meet the Euro 3, Euro 4 and Euro 5 emissions stages for both stoichiometric and lean-burn vehicles. The vehicles selected also cover a wide selection of the European market, with engine capacities ranging from 1.2 to 3.5 litres and power outputs of between 60 and 220 kW.

All vehicles were tested on both the European legislative cycle (NEDC) and the Common Artemis Driving Cycle (CADC) with measurements over the full extent of each of the three CADC elements (urban, extra-urban and motorway) and results will be presented for both cycles. In addition, some of the vehicles were tested from a cold start over the Artemis Urban cycle, which is normally a hot-start cycle, and the effect of this will be identified. Data on each vehicle is available for the three elements of the CADC and for three parts of the NEDC tests (cold-start 1<sup>st</sup> + 2<sup>nd</sup> urban cycles; 3<sup>rd</sup> & 4<sup>th</sup> urban cycles and extra-urban cycle). Some examples of this will be presented together with continuous and cumulative data for particle number emissions.

For the final set of petrol direct-injection test vehicles, PM Number measurements were made using a TSI EEPS (Engine Exhaust Particle Sizer) in addition to the PMP measurement system. This data allows a comparison of the particle number data over the PMP size range (d<sub>50</sub> of 23 nm to 2.5 µm) with EEPS measurements for ranges of both 5 to 500 nm and 20 to 500 nm. In addition, the EEPS allowed an examination of the size distribution of vehicles during the various test cycles.

The results show the variations between petrol-engine technologies over both the legislative and Artemis cycles.

## Effect of residual gas fraction on particle emission on 2/4-stroke direct injection camless engine

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In the automotive industry, gasoline engine downsizing has already been widely accepted as a technology to improving the fuel economy and reducing the CO<sub>2</sub> emissions and it's global warming effects. Direct injection gasoline engines are staging a come-back because of its potential for improved fuel economy through principally the engine down-sizing by boosting or 2-stroke operation, and possibly stratified charge combustion or Controlled Auto Ignition (CAI) at part load operations.

Due to the limited time available for complete fuel evaporation and the mixing of fuel and air mixture, locally fuel rich mixture or even liquid fuel can be present during the combustion process. This causes significant increase in Particulate Matter (PM) emissions from direct injection gasoline engines compared to the conventional port fuel injection gasoline engines, which are of major concerns because of its health implications

An accurate prediction of residual burned gas within the combustion chamber is important to quantify for development of modern engines, especially for those with internally recycled burned gases and HCCI/CAI. A wall-guided GDI engine has been fitted with an in-cylinder sampling probe attached to a fast response NDIR analyser to measure in-situ the cycle-by-cycle trapped residual gas. The exhaust and inlet valve timing were varied for CAI NVO and PVO SI to produce a range of varied lambda and residual gas fractions measured at constant load and speed and the gaseous and particulate emissions were compared. Both strategies reduce the pumping losses and hence increase the efficiency. However the PVO SI can be used as mode transition from SI to CAI combustion at low loads.

In this paper, the effect of residual gas on performance, combustion and emissions are presented for both SI PVO and CAI NVO combustion and the results compared. A wider reduction in specific gaseous emissions was achieved with reduced maximum peak cylinder pressure in CAI NVO. While in PVO SI we have about 93% reduction in particulate emissions compared to CAI NVO within the operational lambda range achieved with the residual gas at engine speed of 1500rpm and constant load of 3.2 bar IMEP<sub>net</sub>.

## Investigation of burning mode for diesel particulate oxidation: contrasting O<sub>2</sub> and NO<sub>2</sub>

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The physical characteristics of diesel particulates from conventional diesel, biodiesel and blends of the two were investigated at several points along the O<sub>2</sub> and NO<sub>2</sub> oxidation reactions. BET measurements elucidated the surface area evolution with extent of oxidation for the two oxidizers and revealed differences in surface area evolution in the samples when oxidized with O<sub>2</sub>. Particulate matter from biodiesel initially has greater surface area than particulate from ULSD, an advantage, which is maintained under O<sub>2</sub> oxidation until approximately 40% of the sample has been consumed. HR-TEM and fringe analysis confirm that initial differences in the lamella lengths have disappeared by 40% burnout of the samples. TEM images suggest that surface area evolves mostly internally for O<sub>2</sub> oxidation as the primary particle diameters do not follow the shrinking core trajectory. However, for NO<sub>2</sub> there are no sample-specific dependencies in either reactivity and surface area. TEM images suggest that NO<sub>2</sub> oxidation occurs by a mostly external trajectory.

## Quantifying aircraft black carbon emissions

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Aircraft emissions of black carbon (BC) contribute to climate forcing, constitute a source of ice nuclei affecting cloud formation and degrade air quality. The smoke number (SN) is the current regulatory measure of aircraft particulate matter emissions and quantifies exhaust plume visibility. Several correlations between SN and the exhaust mass concentration of BC ( $C_{BC}$ ) have been developed, based on measurements relevant to older aircraft engines. These form the basis of the current standard method used to estimate aircraft BC emissions (FOA3) for the purposes of environmental impact analyses. In this study, BC with a geometric mean diameter (GMD) of 20, 30 and 60 nm and filter diameters of 19 and 35 mm are used to investigate the effect of particle size and sampling variability on SN measurements. For BC with 20 and 30 nm GMD, corresponding to BC emitted by modern aircraft engines, a greater  $C_{BC}$  is required to produce a given SN than is the case for BC with 60 nm GMD, which is more typical of older engines. An updated correlation between  $C_{BC}$  and SN that accounts for the typical size of BC emitted by modern aircraft is proposed and an uncertainty of  $\pm 25\%$  accounts for variation in GMD on the range 20-30 nm and for the allowable range of filter diameters. The SN- $C_{BC}$  correlation currently used in FOA3 underestimates by a factor of 2.5-3 for  $SN \leq 15$ , implying that current estimates of aircraft BC emissions derived from SN are underestimated by the same factor. The consequences for estimating global aircraft BC emissions are discussed.

## Bipolar neutralization using radioactive, x-ray, and AC corona methods

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In a typical “neutralizer,” particles pass through a cloud of ions and ideally acquire a steady-state distribution of charge. Commercially available neutralizers utilize radioactive, X-ray, and corona discharge ionization techniques. While there is a common objective to produce equal concentrations of positive and negative ions, these methods differ somewhat. For example, the de-facto standard,  $^{85}\text{Kr}$ , emits highly energetic beta particles. On the other hand, the x-ray charger generates energy x-rays ( $<9$  keV) and the corona discharge is operated with AC voltage.

The objective of this work was to better quantify the impact of neutralization technique and aerosol composition on the neutralization process. Experiments were conducted using solid and semi-volatile, spherical and aggregate particles. Carrier gas composition was varied to examine the sensitivity of charging to aerosol chemistry. Carrier gases included dry nitrogen, humidified air, humidified air with sulfur dioxide, and ambient air. For each neutralizer and carrier gas condition, we measured the ion mobility distribution, particle charged and neutral fractions, and the particle size distribution. These measurements were compared with Fuchs’ theory calculations.

## Aerodynamic aerosol classifier

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A new aerosol particle classifier, the Aerodynamic Aerosol Classifier (AAC), is presented with some of its applications. The instrument uses a centrifugal force and sheath flow between two concentric rotating cylinders to produce a monodisperse aerosol classified by aerodynamic diameter. The instrument is unique in being able to produce a stream of particles of known finite aerodynamic diameter; other technologies such as impactors and virtual impactors provide either an upper or lower cut. Since this instrument does not require charged particles, it produces a true monodisperse aerosol without artefacts caused by multiply-charged particles like other classifiers. This work reports the theoretical and experimental results of the new instrument with some of its applications.

Two diffusion models and two non-diffusion models have been used to predict the performance of the AAC. The limiting trajectory and particle streamline models are analytical methods and do not include particle diffusion. To demonstrate the diffusion effect, a convective diffusion model has been developed by solving the convective-diffusion equation for the AAC which has been solved using the Crank-Nicolson method. The diffusing particle streamline model is an analytical model which models particle diffusion as a Gaussian cross-stream profile about the corresponding non-diffusing particle streamline. The transfer functions were obtained as a function of the particle relaxation time and the particle aerodynamic diameter. The transfer function has been studied for different flow rates. PSL (polystyrene latex) particles and DOS (Dioctyl Sebacate) along with a differential mobility analyzer (DMA) were used to verify the instrument and to obtain the experimental transfer function.

A DMA and the AAC were used in tandem to measure the effective density, dynamic shape factor, and the mass of soot particles emitted from an inverted burner. The measured DMA-AAC mass-mobility exponent was 2.17 for a flame equivalence ratio of 0.67, which agrees well with DMA-CPMA measurements. The effective density was found to vary between 0.18–0.86 g/cm<sup>3</sup> and the dynamic shape factor was calculated to be 1.5–2.6 over the range of 90 to 630 nm in mobility diameter.



## Studies of particulate matter emissions of automotive engines using biofuels

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The control of the PM emissions is expected to commence in European regulation which will require not only the monitoring of PM mass emissions, but also the PM number for all light-duty vehicles. On the other hand, the use of ethanol and other alternative fuels (including bio-fuels) is expected to increase. In this study, the characteristics of PM emissions in a direct injection spark ignition engine are examined with respect to fuel properties and injection strategies. It is shown that the ethanol fraction with dual-injection results in a unimodal mass distribution where the larger, accumulation mode particles are removed. Compared to blends in DI, the dual-injection strategy helps to lower the total PM emissions.