

Overview of Soot Emission Measurements Instrumentation: From Smoke and Filter Mass to Particle Number

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ABSTRACT

Particulate emissions cause adverse health effects and for this reason they are regulated since the 80s. Vehicle regulations cover particulate emission measurements of a model before its sale, known as type approval or homologation. For heavy-duty engines the emissions are measured on an engine dynamometer with steady state points and transient cycles. For light-duty vehicles (i.e. the full power train) the particulate emissions are assessed on a chassis dynamometer. The measurement of particulate emissions is conducted either by diluting the whole exhaust in a dilution tunnel with constant volume sampling or by extracting a small proportional part of the exhaust gas and diluting it. Particulate emissions are measured by passing part of the diluted exhaust aerosol through a filter paper. The increase of the weight of the filter is used to calculate the particulate matter mass (PM) emissions.

Vehicles are also checked during the course of their lives, i.e. those pulled over by authorities for roadside checks or the mandatory periodic inspection in a garage. In this case smoke is assessed with free acceleration tests.

However, all these tests do not give any relevant information about the emissions of the vehicles during real driving conditions. This was only recently regulated. The particulate emissions on the road are measured with portable emission measurement systems (PEMS).

The particulate emissions of newer vehicles decreased by more than two orders of magnitude since the 80s. The particulate measurement methods reached their detection limit and for this reason the particle number (PN) concentration was introduced in the European Union legislation as a new metric.

In this paper a short summary of the particulate emission legislation of vehicles over the years is given. Then the instrumentation that has been used and is currently being used, like gravimetric filter measurement, is explained. In addition, other popular methods and instruments are analyzed, like chemical analysis of filters, light extinction, scattering and absorption instruments, electrical mobility and particle counting instruments. Correlation of the different instruments is discussed for different vehicle emission levels. Predictions for future instruments for low emission vehicles are given.

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INTRODUCTION

Vehicle exhaust particles have long been considered as a significant source of anthropogenically generated particles¹

[1]. Thus, regulation authorities across the globe try to control the levels of emitted particles. Past legislation was initially based upon black smoke levels using opacity meters and over the last 40 years or so, by gravimetric quantification

of Particulate matter Mass (PM) deposited on a filter, from a diluted exhaust sample extracted over a specific test cycle driven on a chassis dynamometer (for light-duty vehicles) or a test bed (for heavy-duty engines) [2].

The gravimetric method reached its limit of detection with the introduction of Diesel Particulate Filters (DPFs). For this reason the Particle Measurement Program (PMP) was launched in 2001 on the initiative of several member states to develop a more sensitive and accurate methodology that would be in position to discriminate the effectiveness of different DPFs [3]. The proposed Particle Number (PN) measurement method from 2009, based upon counting solid (non-volatile²) particles >23 nm was rapidly integrated within the European legislation. Particles <23 nm were excluded in order to avoid the high variability and uncertainty of the nucleation mode particles on many parameters like sampling conditions, facility characteristics etc. Close involvement of the automotive industry and instrument manufacturers in PMP discussions enabled the particle number measurement procedures to be rapidly accessible to legislators, test houses and the automotive industry [4].

A lot of progress has been made for the type approval test procedures regarding PM and PN emissions in order to make them suitable for low emitting engines and vehicles. However, the evaluation of in-service conformity (or in-use compliance in USA) particulate emissions needs new or improved instrumentation [5]. For periodic inspection the devices need to be cheap, small, and light with low maintenance effort. As a result, typically different measurement methods than at the test beds are used; e.g. optical measurement of the smoke with opacity meters.

The latest reviews and instruments comparisons are more than 8 years old [6, 7, 8]. Initially an overview of the legislation will be discussed focusing on the required instrumentation. Then the most commonly used instruments will be shortly presented focusing on their detection limits. Finally predictions for future methods and instrumentation will be given.

LEGISLATION

In this section the legislation of the European Union (EU) will be discussed from the perspective of instrumentation for particulates. There are two types of type-approvals:

- UN/ECE type-approval of vehicle systems and separate components (but not whole vehicles) valid in contracting countries worldwide including the EU.
- EU type-approval of whole vehicles, vehicle systems, and separate components valid in European Economic Area and Switzerland, which is based on Directives and Regulations. In

general, the difference between directives and regulations is that a *directive* requires individual Member States to transpose its requirements into national law to implement it whereas a *regulation* applies throughout the EU without any other action necessary.

There is a strong correlation between the two certifications due to a continuous harmonization process. The EU has already adopted a large number of UN/ECE Regulations and this process will continue until all Regulations are harmonized. As a result, for the products where the two types of legislations (EU and UN) are equivalent to each other, either can be used to market a product in the EU.

Other countries (e.g. United States or Japan) have different emission limits, vehicle categories and test cycles; however the requirements in instrumentation are quite similar. Important differences will be discussed. Other regulations that include particulate emission limits, such as occupational health or aviation, will not be a focus in this paper. Detailed information for legislation topics can be found in [9, 10].

Type Approval

The type approval for many gaseous pollutants, PM and PN is conducted with the Type I test, i.e. verifying the average emissions after a cold start, conducted over the New European Driving Cycle (NEDC). Smoke emissions are also checked but with a different procedure.

Smoke

Smoke³ emissions of diesel engines for use in vehicles were controlled from the 70s [11] (and last time amended by Directive 2005/21/EC [12]). The diesel engine submitted for approval is measured by two methods: test at steady-speed over the full-load curve, and test under free acceleration. The emissions measured according to the steady-speed test have to be lower than specific limit values, as described in Regulation 24 [13].

Peak opacity of Heavy-duty Diesel exhaust emission was also limited in the European Load Response (ELR) test, defined in the Euro III Heavy-duty Regulation [14] until Euro V. For Euro VI engines the smoke test and limits are not applicable due to the introduction of the PN type approval test. In contrast, the US Smoke test [15] sets opacity limits for acceleration, lug down and steady state during a composite speed/load pattern.

¹The term *particle* is conventionally used for the matter being characterized (measured) in the airborne phase (suspended matter), while *particulate matter* refers to the collected material on a sampling filter, according to the regulations.

²In this paper *non-volatile* particles or solids are defined those that do not evaporate at approximately 350°C. Those that do evaporate (volatiles and semi-volatiles) are defined as *volatile* particles.

³*Smoke* refers to particles, either solid or liquid, suspended in the exhaust stream, which interact with light. Diesel exhaust smoke can be white (water), blue (unburnt fuel and lubricant) or black (soot).

PM Method

Directive 88/436/EEC [16] introduced limit values and a procedure for measuring particulate emissions of light duty vehicles equipped with Compression Ignition (CI) engines. Directive 91/542/EEC [17] introduced Euro I and Euro II standards in 1992 and 1996 respectively for heavy-duty engines, including both the gaseous and, for the first time, the particulate emissions. The Euro 5/6 (light-duty) [18, 19] and Euro VI (heavy-duty) [20, 21] implementing legislation introduced an improved PM emission measurement method developed by the United Nations - Economic Commission for Europe (UN/ECE) Particulate Measurement Programme (PMP), also adjusting the PM emission limits to account for differences in results using the old and the improved method.

The gravimetric PM method uses, a probe sampling from a dilution tunnel, a filter unit, a partial-flow pump, a flow regulator and a measuring unit. The PM mass of the test is calculated by using the mass collected on the filters (primary and back-up until Euro 4) and the volumes of the exhaust flowing through particulate filters and the diluted exhaust gases. Since Euro 5/6 the temperature of the sampled diluted exhaust gas is controlled with more precision (with some more additions and improvements) and only one filter is used.

PN Method

The Euro 5/6 (light-duty) and Euro VI (heavy-duty) [18, 21] legislation introduced a PN emission limit and measurement procedure, in addition to the mass-based limits. The particle sampling system consists of a probe sampling from a dilution tunnel, a Volatile Particle Remover (VPR) upstream of a Particle Number Counting (PNC) and the transfer tubing. The VPR should dilute the sample by a factor of at least 10, and heat it at a temperature of $\geq 150^{\circ}\text{C}$ and $\leq 400^{\circ}\text{C}$. The volatile particles are evaporated in the Evaporation Tube (ET), which should be controlled at a wall temperature between 300 and 400°C . The gas sample should be diluted to achieve a PN concentration below the upper threshold of the PNC and a gas temperature below 35°C at the PNC inlet. The PNC, which is a Condensation Particle Counter (CPC), must have counting efficiencies at particle sizes of 23 nm and 41 nm electrical mobility diameter of 50% and >90% respectively.

For light-duty vehicles the PN limit of 6.0×10^{11} p/km of the Type I test is applicable to all Euro 5b CI vehicles (September 2011). Euro 6 Positive Ignition (PI) vehicles with Direct Injection (DI) engines must comply with the same PM and PN limits as CI vehicles. However, in order to allow the development and implementation of all necessary technologies that allow the vehicles to meet the CI vehicles PN limit, Commission Regulation 459/2012 [22] has adopted a two-step approach: Until three years after the Euro 6 implementation dates a particle number emission limit of 6.0×10^{12} p/km shall apply to Euro 6 PI DI vehicles upon the choice of the manufacturer. In addition, until those dates at the latest (3 years after Euro 6 implementation) a type

approval test method ensuring the effective limitation of particles emitted by vehicles under real driving conditions shall be implemented [22].

For Euro VI heavy-duty CI engines limits of 8.0×10^{11} p/kWh and 6.0×10^{11} p/kWh over the Worldwide Harmonized Steady state Cycle (WHSC) and Worldwide Harmonized Transient Driving Cycle (WHTC) respectively were introduced [18]. For the respective heavy-duty vehicles equipped with PI engines fuelled with natural gas or liquefied petroleum gas, PN limit over the WHTC shall be introduced at a later stage.

In-Service Conformity

In 2001 (Euro 3) a conformity test was introduced in order to confirm the functionality of the emission control devices during the normal useful life of the (light-duty) vehicles under normal conditions of use. Type I test and test for On-Board Diagnostics (OBD) are subjected to in-service conformity test [19]. A number of vehicles are tested and after a statistical analysis of the results. The type approval of Vehicle types that failed the tests is withdrawn.

The in-service conformity testing of Euro IV/V (heavy-duty) engines is conducted as follows: An engine from a vehicle in normal use is tested over the European Stationary Cycle (ESC) and the European Transient Cycle (ETC) tests for gaseous and particulate emissions and over the ELR test cycle for smoke emissions. On the request of the manufacturer the approval authority that granted the original type-approval may decide to use the in-service conformity procedure for Euro VI vehicles, using Portable Emissions Measurement Systems (PEMS) [18] on the road over their normal driving patterns, conditions and payloads.

Although the PEMS testing is applicable for both gaseous and PM emissions in the USA [23], in Europe it is currently applicable to gaseous emissions only. In 2008 European Commission launched the EU-PEMS PM evaluation program, to assess the potential of portable instruments to measure particulate emissions on-board of vehicles [24, 25, 26].

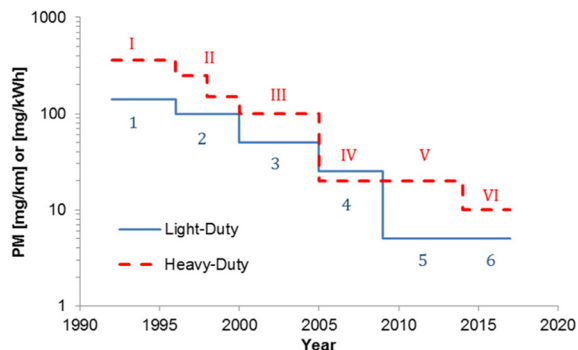


Figure 1. Evolution of PM type approval emission limits for CI light-duty passenger cars (M_1) and heavy-duty engines. Numbers indicate Euro levels.

Figure 1 shows the evolution of Type I test PM emission limits in Europe for CI passenger cars and heavy-duty engines. The limits have been reduced by almost 2 orders of magnitude.

Roadworthiness Test

The road worthiness test for passenger cars was firstly introduced by Directive 77/143/EEC [27], in order to harmonize the frequency of checks and the compulsory items to be tested among the Member States. To guarantee that the vehicles do not exceed the defined emission limits throughout their life time, the exhaust emissions were added to this periodic inspection test with Directive 92/55/EEC [28], applicable to both light and heavy-duty vehicles. With Directive 96/96/EC [29] the applicable tests, including exhaust emission tests, were consolidated in a single text. For vehicles equipped with PI engines a measurement of the carbon monoxide in idle speed was required. In the case of vehicles equipped with CI engines the measurement of exhaust gas opacity with free acceleration (no load acceleration from idle up to cut-off speed) is applicable. The test is conducted according to Directive 72/306/EEC [11] with an opacity meter measuring at the exhaust gases emitted by the vehicle. With Directive 2003/27/EC [30] the limit values were reduced for Euro 4 and Euro IV light and heavy-duty vehicles respectively, in order to take into account the technical improvements of such technology engines. Directive 96/96/EC [29] and all its amendments were recently consolidated in Directive 2009/40/EC [31], as updated by Directive 2010/48/EU [32].

The free acceleration smoke test is used also in non-EU countries with the exception of some locations such as Australia and the US where loaded tests (on a simple chassis dynamometer) are conducted typically at steady states or even at transient modes in some cases. The periodic technical inspection of in-use vehicles is sometimes part of bigger Inspection and Maintenance (I&M) programs, where also roadside inspection is conducted. I&M programs are often maintained by lower level governments, such as by states in the United States or by provinces in Canada.

INSTRUMENTATION

There are many instruments that are used to measure engine exhaust gas particles and information can be found in text books [e.g. 33, 34, 35, 36, 37]. Quite often instruments' reviews [8, 38, 39, 40] or comparisons [e.g. 38, 4041, 41, 42, 43] are done. A summary of the most commonly used instruments in the automotive field follows. The focus is given on the instruments that are mature enough for the near future with detection limit sufficient for modern vehicles with DPFs.

Commonly Used Instruments

Table 1 presents the most commonly used instruments in the automotive field classified by their principle of operation.

It gives the particle property that is being measured (mass, surface area, number), whether it is a real time instrument (i.e. whether it measures with a resolution of at least 1 s) and whether it can give size information (e.g. a mean size or a size distribution). The instruments are separated in two categories: i) collection and analysis and ii) direct-reading sensors.

Table 1. Most commonly used methods and instruments in the automotive field. M=mass, N=number, S=surface.

Principle	Name	Property	Real time / Size info
FILTER METHOD			
Weighing	PM	M	No / No
CHEMICAL ANALYSIS			
Dissolution	Soxhlet extraction	PM=SOL+SOF+SUL	
Gasification	Thermodesorption	PM=NVF+VF	
	TGA	PM=SUL+VF+EC+Ash	
	Thermochemical	PM=EC+OC	
OPTICAL METHODS			
Extinction	Opacity meter	M (opacity)	Yes / No
	Spot meter	M (FSN)	No / No
	Aethalometer	M (BC)	Yes / No
Scattering	Phototmeter	M (Smoke)	Yes / No
	Nephelometer	M (Smoke)	Yes / Yes
	OPC	N	Yes / No
	CPC	N	Yes / No
Absorption	PASS	M (BC)	Yes / No
	LII	M (BC)	Yes / Yes
MICROBALANCE INSTRUMENTS			
Frequency	TEOM	M	Yes / No
	QCM	M	Yes / No
CHARGING METHODS			
Radioactive	Epiphaniometer	S	No / No
	Beta gauge	M	No / No
Electrical	Diffusion charger	S	Yes / No
UV light	Photoelectric	S	Yes / No
COMBINED SIZE DISTRIBUTION METHODS			
Impaction	Impactors	M	No / Yes
Optical	Microscopy	N, S	No / Yes
Charging + Classifying + Counting	CMPA	M	No / Yes
	EAA	N	No / Yes
	DMA, SMPS	N	No / Yes
	DMS, EEPS	N	Yes / Yes
	FIMS	N	Yes / Yes
	ELPI, DMM	N, M	Yes / Yes
	EDB	N	Yes / Yes