

Evaluation of PM Measurement Precision and the Equivalency of the Single and Three Filter Sampling Methods for LEV III FTP Standards

Satya Sardar, Sherry zhang, Larry Larsen, Bruce Frodin, Wayne McMahon, and Shiou-Mei Huang
California Air Resources Board

M.-C. Oliver Chang
California Air Resource Board

ABSTRACT

Present motor vehicle particulate matter (PM) emission measurement regulations (Code of Federal Regulations (CFR) 40 Part 1065, 1066) require gravimetric determination of PM mass collected onto filter media from dilute exhaust. To improve the current sampling and measurement procedures for TIER 3 PM emissions standard of 3 mg/mile, CFR part 1066 adopted five alternative PM sampling options. One option of great interest is sampling the entire test using a single flow-weighted filter rather than the conventional three-filter (one filter per test phase) approach. The single filter method could lessen the time needed for gravimetric determination by reducing the quantity of filters used for a test and possibly reduce the uncertainty in gravimetric measurements, particularly at sub 1 mg/mile PM levels. This study evaluates the single filter and, to a limited extent, the 2-filter alternatives adopted in 40 CFR Part 1066. The first part of this study comprised evaluation and equivalence assessment of a Horiba Quad PM sampler along with an AVL 472 SPC PM sampler. PM emission data were collected at Air Resources Board's Haagen-Smit Laboratory (HSL) over Federal Test Procedure (FTP), Urban Dynamometer Driving Schedule (UDDS) and US06 cycles for twelve vehicles. Measurement precision for both samplers was determined to be 11% for PM loadings in the 10 to 60 μg range. Furthermore, statistical analysis determined that the majority of CFR-compliant PM sampling instruments are also likely to have measurement precision close to 11%. The second part of the study assessed the equivalency of PM emission measurement using the single and 2-filter sampling options in conjunction with the conventional 3-filter method. A total of eight vehicles, which comprised a subset of the vehicles tested for the evaluation of the sampler equivalency and estimation of measurement precision, were tested over the FTP and UDDS cycles. Two test vehicles had FTP PM emissions of approximately 3 to 4 mg/mile and six vehicles had emission levels below 1 mg/mile. Results for the single filter method, when compared with the conventional 3-filter method, indicated very high correlation with R^2 close to 0.99 and 0.92 for PM emission levels below 3 mg/mile and sub 1 mg/mile, respectively. The data also indicated that the single, the 2-filter, and the 3-filter methods had similar variability of measurements irrespective of the vehicle's emission level. Interestingly, the single filter method yielded slightly lower (less than 10%) PM emission compared to the 3-filter method. The difference is primarily attributed to background contributions (three vs. one) and/or likely the loss of the semi-volatile fraction of PM from the single filter because it is subjected to a vacuum environment for a longer period of time as compared to the conventional 3-filter method. Nevertheless, tunnel blank corrections demonstrated better correlation and convergence of data between the two methods. Statistical analysis (t-test) indicated no significant difference between the single and the conventional 3-filter method. The single filter method has the prospect of alleviating measurement challenges at the 1 mg/mile level to support implementation of the LEV III standards.

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INTRODUCTION

Ambient particulate matter (PM) has received significant attention because numerous epidemiological and toxicological studies have indicated its potential for eliciting adverse health effects [1, 2, 3, 4, 5]. PM has also been associated with detrimental impacts on the air quality, visibility, and climate [6].

Vehicular emissions constitute a major source of urban PM pollution both due to direct emissions, via combustion and mechanical wear, and secondary formations, whereby organic and inorganic vapors undergo photochemical reactions and gas-to-particle conversion in the atmosphere [7, 8]. Vehicular emissions thereby contribute significantly to human exposures especially in sites with higher PM levels, for example, the on-road environments and near freeway locations [9, 10, 11].

To ensure vehicle PM emissions are well-controlled, the California Air Resources Board (CARB) adopted the Low Emission Vehicle III (LEV III) regulation for light-duty vehicles (LDV) in 2012. The LEV III regulation encompasses a phase-in reduction of the current FTP PM emission standard of 10 to 3 mg/mile over the period of 2017 to 2021 and finally, a phase-in of a more stringent 1 mg/mile standard from 2025 to 2028 [12]. The U.S. EPA has adopted the Tier 3 program, which similarly lowers PM emission standards for LDVs to 3 mg/mile beginning with model year 2017 [13].

PM emission standards are expressed in mass of PM emitted per unit distance traveled. Present motor vehicle PM emission measurement regulations (40 CFR Part 1065, Part 1066) [14, 15] require gravimetric determination of PM mass collected onto a filter media, from dilute exhaust, maintained at a filter face temperature of 47 ± 5 °C. With the current sampling and measurement procedures, PM mass collected for a 1 mg/mile vehicle could be in the range of 10 to 100 µg per filter depending on the vehicle, test cycles, or the phase. At these low PM emission levels, concerns have been expressed about challenges with the current gravimetric method such as measurement variability. Moreover, the filters are susceptible to gaseous adsorption, predominantly organics, which can contribute 5 ± 10 µg weight gains even in the absence of a test vehicle when an undesirable media is used for sample collection [16]. This gaseous artifact was acknowledged at the time of the 2007 revisions to the PM measurement methodology, which formed the foundation for CFR Part 1065 modifications to the gravimetric method [17, 18]. This artifact could be the cause of some measurement uncertainty for vehicle emission testing. Moreover, semi-volatile organic compounds (SVOC) are also a part of LDV PM emissions [19]. The SVOCs are also significantly influenced by the sampling conditions (e.g., dilution process, temperature, relative humidity) [20, 21]. These SVOCs and volatile organic compounds (VOC) can contribute to both positive (adsorption) and negative (evaporation of volatile components) mass, resulting in some biases in emission calculations at low PM emission levels [21]. These artifacts are substantially minimized with the usage of Polytetrafluoroethylene (PTFE) filters for PM collection. However, the above mentioned artifacts are not completely eliminated with the use of PTFE filters and could still affect PM emission results.

Recently, CARB and U.S. EPA demonstrated measurement capability of PM emissions below 1 mg/mile using the existing filter-based (3-filter) gravimetric method [22]. However, alternative measurement approaches are also under evaluation [15] to alleviate some of the aforesaid challenges. For instance, Bushkuhl et al., have attempted to measure low PM emissions by combining the gravimetric filter approach along with real-time measurements [16]. Another study reported by Yang Li et al., involved evaluation of the integrated particle size distribution (IPSD) method and size dependent particle density in comparison with the current gravimetric method for measuring PM emissions from light duty vehicles [23]. The abovementioned studies are based on both real-time instruments and particle size distributions, whereas this paper focuses on using the existing regulatory gravimetric definitions and test procedures for measuring PM at the levels required by the LEV III PM standards.

CFR Part 1066 [15] defined five different PM sampling options for exhaust emission test procedures using the FTP test cycle. The single filter (option 4) and 2-filter method (option 2) options are of great interest because they can decrease the number of filters needed for a FTP test, thereby, reducing the time and resources needed for gravimetric determination. Furthermore, filter compositing as proposed by the single filter method can also improve the sensitivity of measurement by reducing the total uncertainty related to weighing and handling of three filters.

The first part of this study comprised evaluation of a Horiba Quad PM sampler with a collocated AVL 472 SPC PM sampler. The Horiba PM sampler has a common exhaust inlet from the Constant Volume Sampler (CVS) dilution tunnel which subsequently divides into four different collection streams. Equivalence of the four Horiba streams and the SPC sampler was established. The equivalence of the PM measurement instruments was aimed to confirm that there is no natural bias of any of the units which could lead to erroneous results while comparing the single, 2-filter and the 3-filter methods. Measurement precision was also determined at low PM emission levels for the two samplers with an aim of determining a universal number applicable to CFR-compliant PM sampling instruments. The second part of this work evaluated equivalence of some alternative sampling methods proposed in CFR Part 1066. Specifically, the flow weighted single filter and full flow 2-filter methods are compared to the conventional 3-filter method. However, one cautionary note for the single filter method could be the impact of a higher sampling flow rate in Phase two (vacuum) of the FTP cycle as compared to phase one and three, which might possibly result in loss of the semi-volatile fraction of PM. This impact needs to be evaluated in future studies.

EXPERIMENTAL SETUP

This section describes the procedures used in CARB's HSL in El Monte to establish PM measurement equivalency between the Horiba Quad and AVL 472 SPC PM samplers and to compare single filter, 2-filter, and the conventional 3-filter PM measurement approaches as adopted in 40 CFR Part 1066 [15].

Test Vehicles and Fuel Used

In this study, a total of twelve vehicles were tested including two GDI and ten PFI vehicles. These vehicles were involved in various studies and hence, data presented in this paper encompasses numerous research projects. The vehicles were selected for FTP emission rates below 3 mg/mile and, predominantly, below 1 mg/mile, representative of the forthcoming LEV III standards. The first part of this study, which investigates PM sampler equivalency (Horiba Quad & AVL 472 SPC), used data from all twelve vehicles listed in [Table 1](#).

The second part, which explores the single filter, 2-filter, and the 3-filter methods, used data from a subset of the vehicle pool. Specifically, vehicles 1 to 8 were used for evaluation of the single filter vs. 3-filter method and vehicle 5 was used for 2-filter and 3-filter comparison. Furthermore, most of the vehicles were tested using California Phase III certification gasoline with the exception of vehicles 6 and 7, which were tested using EIA 150 fuel.

Table 1. Vehicle characteristics

Veh No.	Make/ Model	MY	Vehicle Category	Tech	Odometer (K, miles)	Emission Category
1	Honda Civic	2012	PC	PFI	32	PZEV
2	Chevy Malibu LT	2012	PC	PFI	26	PZEV
3	Toyota Camry LE	2009	PC	PFI	67	SULEV
4	Dodge Grand Caravan	2013	LDT	PFI	40	SULEV
5	Nissan Altima	2011	PC	PFI	30	PZEV
6	Buick Regal	2011	PC	PFI	11	ULEV
7	Ford Explorer	2012	LDT	PFI	21	ULEV
8	Honda Accord	2012	PC	PFI	32	PZEV
9	Chevy Silverado	2007	LDT	PFI	158	ULEV
10	Ford Fusion	2013	PC	GDI	9	SULEV
11	Nissan Juke	2011	PC	GDI	51	ULEV
12	Smart Fortwo	2008	PC	PFI	77	ULEV

Test Cycle

For the Horiba Quad and AVL SPC 472 PM sampler equivalence evaluation, the vehicles were tested over the FTP and US06 cycles. For the second part, PM samples were collected by the Horiba sampler to evaluate the single filter, 2-filter, and 3 filter methods. During this part, the vehicles were tested predominantly over the FTP cycle and some, over the double Urban Dynamometer Driving Schedule (UDDS) cycle. More specifically, the double UDDS cycle was selected for the comparison of the 2-filter and 3-filter methods and the FTP cycle was selected for the evaluation of the single filter vs. 3-filter approaches. The double UDDS cycles were run with a 10 minute hot soak in between and one PM sample collected for each UDDS cycle. For the FTP cycle, three separate filters collected PM samples corresponding to the three phases of a regular FTP cycle or a single filter was employed when the flow-weighted approach was used.

Experimental Setup

Figure 1 shows a schematic of the sampling system. Test vehicles in HSL were tested on a 48" single-roll AC electric dynamometer and a CVS system with a 10-inch diameter full dilution tunnel. The CVS system maintains a constant flow of the exhaust and the dilution air blend. Vehicle exhaust emissions were transferred through a 1.5 meter stainless steel transfer tube to a mixing point of the tunnel where vehicle exhaust was combined with dilution (ambient) air. The dilution air was scrubbed first through a charcoal bed to reduce HC and SVOC background concentrations and then passed through HEPA filters with greater than 99.97% removal efficiency. The nominal CVS total flow rates were 350 standard cubic feet per minute (scfm) for the FTP and UDDS cycles, and 750 scfm for the US06 cycle. Sample probes for PM measurement were located near the

tunnel center line and more than ten tunnel diameters downstream of the point where dilution air mixes with tailpipe exhaust. Weekly quality control checks were performed to ensure compliance with CFR requirements for the CVS system, gas analyzers, and PM sampling instruments (40 CFR Part 86 for testing and Part 1066 for PM sampling). Real-time instruments were also used to collect total particle number (PN) using a Grimm CPC (Model 5.400), size distribution data using a TSI EEPS (Model 3090), and black carbon from an AVL 483 Micro Soot Sensor (MSS), depending on instrument availability. Results for the PN counts, size distribution, and black carbon measurements will be discussed and presented in separate peer-reviewed manuscripts.

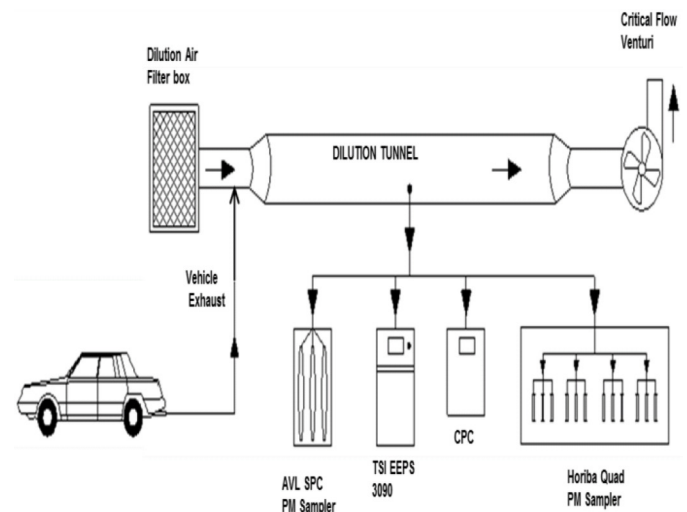


Figure 1. Schematic of test setup

PM Sampling

PM emissions were sampled using an AVL SPC 472 (Anstalt für Verbrennungskraftmaschinen, Germany) and a Horiba Quad sampler (Japan). The quad sampler has a 1-inch outer diameter (OD) probe from the CVS tunnel which subsequently splits out into four PM sampling units (OD of 0.63 inch). The four PM units have dedicated pumps for sample flow. The probe OD and ID for the SPC PM Sampler are 0.625 and 0.5 inch respectively. Both sampling units are designed to meet the PM sampling requirements in CFR Part 1066 [15]. Each PM sampler is equipped with a cyclone pre-classifier to eliminate particles above 2.5 μm in aerodynamic diameter. PM samples were collected on 47 mm diameter PTFE Teflon filters (2 μm , Whatman, USA). For the SPC sampler, the filter flow rate was set at 60 liter per minute (lpm), and the Quad sampler collected samples at a targeted flow rate of 60.5 lpm. For both samplers, the filter face temperature was maintained within $47 \pm 5^\circ\text{C}$ for all tests.

Before testing, the filters were equilibrated for more than an hour both before and after test weighing in the ISO 14644-1 Class Six cleanroom environment. The room temperature was maintained at $22 \pm 1^\circ\text{C}$ and the dew point was sustained at $9.5 \pm 1^\circ\text{C}$ (SOP MV-AEROSOL-145) [24]. Details on the filter handling procedure can be found in SOP MV-AEROSOL- 155 [25]. Static charge on filters was neutralized by Po210 α -particle neutralizers installed in the weighing chamber. Filter weighing is conducted by a robotic AVL (Measurement Technology Laboratories) Filter Weighing System.

(FWS). More precisely, a high precision microbalance (Mettler-Toledo Inc., Columbus, OH), with 0.1 μg readability and 0.25 μg repeatability for the NIST traceable metal weight standard, was used for filter weighing. For both PM samplers, tunnel blanks were collected at least weekly. For tunnel blank sampling, the vehicle exhaust transfer tube was sealed. The duration of the test, CVS flow rates and PM sampling parameters were the same as an emission test.

RESULTS AND DISCUSSION

Equivalency Evaluation of the Horiba Quad and AVL SPC 472 Sampler Units and Measurement Precision

Correlation of PM Emissions (mg/mile)

The first phase of this study investigated the equivalency of the AVL 472 SPC and the four Horiba Quad sampler units. As noted earlier, twelve vehicles with emissions below 3 mg/mile were tested as part of this analysis. A total of 86 FTP and US06 tests were conducted. Most of the data presented for the equivalency evaluation is based on weighted FTP cycle results using the conventional 3-filter method and the flow weighted FTP method as described in 40 CFR Part 1066. However, for a few cases, only phase 1 PM mass is sampled and used for this correlation. This analysis also includes some US06 test data. Of the 86 tests, only 41 tests had collocated data for all four units of the Horiba Quad sampler and the AVL SPC sampler.

The data collected for the 41 valid tests were further subdivided into three bins based on the emission levels, namely, above 3 mg/mile, between 1 and 3 mg/mile, and below 1 mg/mile. Figures 2a and 2b present the emission rates for the 35 tests with emissions below 3 mg/mile. In addition to emission rate, for every individual test, the average, one standard deviation, and coefficient of variation (COV) are also presented immediately to the right of the test data. Figure 2a displays the comparison between the Horiba and AVL samplers for the emission range of 1 to 3 mg/mile. As can be observed from Figure 2a, PM measurements are comparable for all five units in this range. The COV is found to be less than 10% for all of the tests conducted with the exception of Test Number 3.

The six tests with PM emissions greater than 3 mg/mile, the majority of which consisted of tests where only phase 1 of the FTP was sampled, are not shown here. However, for these tests, all five sampler units have shown very high correlation with COV of less than 6%.

Figure 2b compares PM emissions for the sub 1 mg/mile range. The emissions measured by the five units have COV less than 20% for most of the tests. Furthermore, an inverse relationship between PM emission level and COV is observed for the five units. For example, for test numbers 8, 9, and 10 with PM levels close to 1 mg/mile, the COVs are less than 5%. However, the COVs are slightly higher, between 10% to 20%, for most of the tests with emissions below 0.5 mg/mile. For this dataset, the COV ranged between 2.9% to 21.0% with an average of 12.5% for the sub 1 mg/mile level.

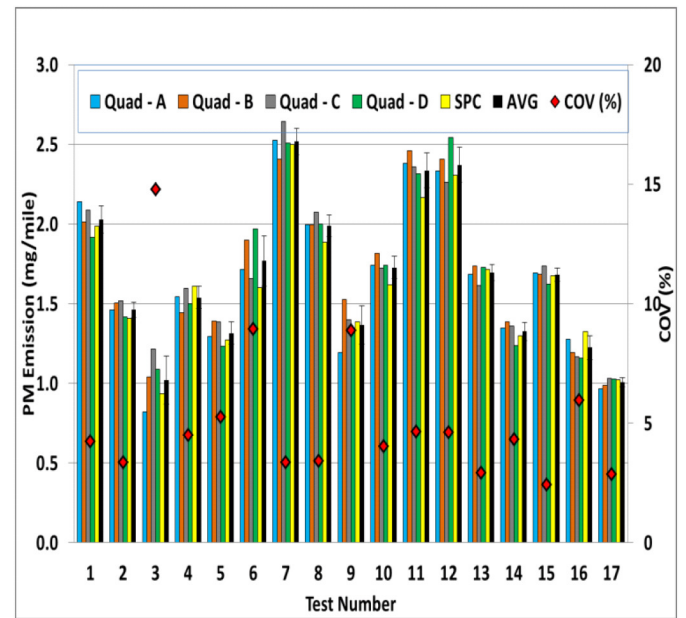


Figure 2a. Horiba Quad and AVL 472 SPC Sampler comparison for 3 to 1 mg/mile range

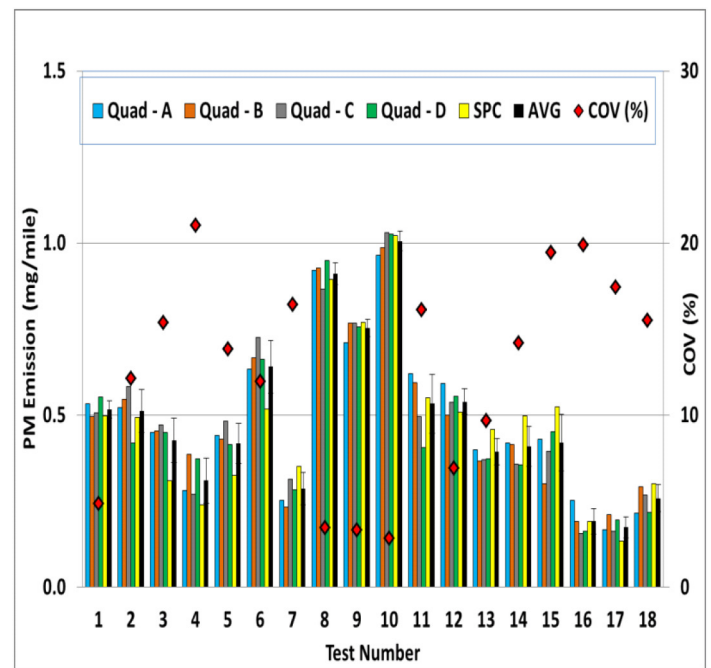


Figure 2b. Horiba Quad and AVL 472 SPC Sampler comparison for sub 1 mg/mile range

Correlation of PM Loadings (μg)

PM loading data from 86 tests were compared to correlate gravimetric measurements between samplers, using the SPC as a reference unit. The raw PM mass was examined to more accurately reflect the signal to noise ratio in gravimetric determination. Although the PM mass was not corrected for the corresponding flow rate, the correction was minor (COV < 0.01% between flow rates) as the flow met the CFR criteria.