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Impacts of Exhaust Transfer System Contamination on Particulate Matter Measurements

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Abstract

As the measurement of low levels of particulate matter (PM) and solid particulate number (PN) from light-duty vehicles becomes more critical, it is becoming more important to understand the potential impacts of exhaust transfer system contamination. This phenomenon occurs when, as it relates to vehicle emission testing, particles deposit and semi-volatile materials are adsorb onto the inner surfaces of the exhaust transfer system, which includes the vehicle exhaust pipe, the exhaust transfer line, and the constant volume sampling (CVS) system, and may subsequently re-entrain and desorb in subsequent vehicle tests. A soot loading cycle was successfully developed and resulted in 36 to 8600 mg of PM mass passing through the CVS tunnel. The results from cleaning tests suggested that majority of particles released during the cleaning tests are semi-volatiles with little presence of soot. A series of chassis dynamometer tests were conducted to characterize the differences between "clean" and "contaminated" sampling system and their impact on low level PM measurements. The results from this study show no measurable PM mass impacts between the "dirty" and clean tunnel conditions that were observed until after a high emitter was tested (80–120 mg/mi diesel vehicle).

1 Introduction

Particulate matter (PM) is a common air pollutant that has been associated with multiple adverse health outcomes [1]. Light-duty vehicles (LDVs) represent approximately 10% of the PM emissions inventory for mobile sources in the United States of America (USA) [2]. LDVs with gasoline port fuel injection (PFI) engine configurations have typically had relatively low PM emissions, but the light-duty vehicle (LDV) fleet is rapidly transitioning to more gasoline direct injection (GDI) vehicles due to their better fuel economy and correspondingly lower CO_2 emissions. GDI engines began market penetration for cars in 2007 and for light trucks in 2008. By 2015, the market share for GDI had grown to 50.7% for cars and 43.2% for light trucks [3]. Furthermore, the GDI vehicle market share is projected to grow around 10% each year to eventually dominate the light-duty market [4]. Unfortunately, the combustion process for GDI engines inherently generates more PM emissions than its PFI counterpart [5, 6].

The expected growth of GDI engines in the LDV market has led to the introduction of more stringent PM standards for LDVs in the USA. PM standards were reduced from 10 to 3 mg/mi in 2017 as part of the United States Environmental Protection Agency (EPA) Tier 3 and the California Air Resources Board (CARB) Lower Emission Vehicle (LEV) III regulations, with a further reduction to 1 mg/mi in 2025 included as part of the California LEV III requirements [7, 8]. For Europe, tighter particle standards have been implemented through solid particle number (SPN) limits, with the Euro 6 standard for light-duty diesel vehicles set at 6.0×10^{11} #/km as of 2014, with a looser standard of 6.0×10^{12} #/km for positive ignition (or spark ignition) LDVs until 2017 [9].

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An important issue with the implementation of these new PM standards is the challenge associated with the accurate measurement of PM emissions at such low levels. Several studies have investigated the challenges of PM sampling and measurement at low levels for LDVs [10–12]. These have included studies to evaluate different methodologies to better quantify the light-duty PM mass emissions through lowering CVS dilution ratios, combining filters, and increasing filter face velocity (FFV). The use of partial flow device (PFDs) as an alternative to the constant volume sampling (CVS) system for PM sampling method has also been investigated [10, 13–19]. PFDs are of particular interest to quantify very low PM mass because of their potential to reduce the artifact from exhaust transfer system, such as exhaust transfer line and CVS. While the surface to volume ratio is higher for PFD, it is much easier to maintain the surface of the PFDs mini tunnel clean leading to less deposition/adsorption artifact. More importantly, PFDs normally would take samples upstream of the transfer line, which would reduce the potential impacts of resuspension of deposits and vapors (mainly organic) from the transfer line walls [14, 15].

Another important aspect of newer certification procedures is that they include PM standards for more aggressive cycles than have been used in the past. The EPA Tier 3 and CARB LEV III regulations have established a PM standard of 6 mg/mi for the US06 portion of the Supplemental Federal Test Procedure [7, 8]. Maricq et al. [20] and Xue et al. [21] found higher PM mass during more aggressive cycles for the gravimetric PM measurement method compared to other aerosol measurements due to higher sensitivity of the gravimetric method to semi-volatiles, which are emitted at higher levels during aggressive cycles. Europe, China, and many other countries throughout the world have implemented or are moving to adopt the Worldwide harmonized Light vehicles Test Procedure (WLTP) into their emissions certification procedures, which includes an extra high speed portion with vehicle speeds up to 130 km/h and higher accelerations [20]. These high speed cycles are intended to represent real world driving that occurs on highways and the emissions that may occur under such driving conditions.

While measuring PM at low emission levels presents challenges, the measurement of PM over more aggressive/higher speed cycles can also introduce additional challenges. Engine operation at higher speeds and loads results in high exhaust temperatures. The increase in the exhaust temperature will produce changes in the nature of particle emissions relative to the better understood US Federal Test Procedure (FTP) and EU New European Drive Cycle (NEDC). Additionally, the high exhaust temperatures can lead to storage/release of materials from the vehicle exhaust system and exhaust sampling system that interfere with particle measurements [22, 23]. Exhaust transfer/sampling system contamination, as it relates to vehicle emission testing, can occur as a result of deposition of particles and adsorption of semi-volatiles onto the inner surfaces of the exhaust transfer system, which can include the vehicle exhaust pipe and exhaust transfer line in connected with a traditional CVS system. These deposited particles can reentrain to the exhaust stream to some extent, while the adsorbed semi-volatiles can desorb from the exhaust transfer line during subsequent vehicle chassis dynamometer certification cycles or under real driving emissions (RDE) conditions, generating additional PM mass that could impact the vehicle certification progress, particularly with respect to PM. This contamination can be significant, especially as the LDV emission levels continue to be reduced, so it is important to better characterize and understand how it might impact PM emissions measurements.

The objective of the present study is to better characterize the potential impact of contamination in the sampling system during low level PM emission measurements. This is the first major effort to systematically evaluate these contamination impacts. For this study, a series of chassis dynamometer emissions tests were conducted to identify and quantify the sources of contamination from the exhaust sampling system. The test cycles included the FTP, US06, and a "cleaning" cycle that was conducted with a natural gas burner. PM sampling was done with both a conventional CVS and a PFD measuring in the raw exhaust. Some goals of the present study were to identify when desorption from the exhaust transfer system occurs during aggressive driving cycles, to better understand the nature of the PM generated during contamination events, and to understand how this contamination related PM impacts vehicle PM measurements. Another issue examined in this work is how PFDs perform when higher exhaust temperatures occur during vehicle testing and how PFDs correlate with the CVS PM sampling under such conditions. Overall, the results of this study demonstrate that aggressive drive cycles pose important challenges to particle sampling for vehicle certification testing.

2 Experimental Section

2.1 Test Vehicles

A total of 4 test vehicles were used to provide a wide range of PM emissions. This included a mix of vehicles with very low emission rates coupled with higher emitting vehicles to contaminate the sampling system. Descriptions of the test vehicles are provided in Table 1. A 2012 PFI vehicle with a PM emission rate well below 1 mg/mi over the FTP cycle was used as a reference vehicle for this test. It is denoted as PFI_L. This vehicle was tested before and after the tunnel contamination was conducted to determine the extent to which contamination would impact PM emission rates.

Table 1 Test vehicle descriptions

MY	Certification	Type	Engine size	Mileage	Designation	FTP PM emission rate (mg/mi)
2012	LEV2 ULEV PC	PFI	1.6 L	5509	PFI_L	< 1
2016	ULEV II PC	GDI	1.6 L	5485	GDI_H	3
2014	Mexico	Diesel	2.0 L	31,352	Diesel_H1	80
2016	Mexico	Diesel	2.0 L	4785	Diesel_H2	20

GDI = gasoline direct injection, *PFI* = port fuel injection

Three vehicles were utilized as high emitter PM sources that could potentially contaminate the CVS tunnel and sampling systems. This includes a 2016 GDI vehicle (GDI_H), a 2014 diesel vehicle (Diesel_H1), and a 2016 diesel vehicle (Diesel_H2). GDI_H, which had much higher PM emissions (~ 3 mg/mi over the FTP) than the reference vehicle, which was used as the initial contamination source for this testing. After conducting two repeats of the test sequence provided in Table 2, and using the soot loading cycle discussed below, a third test sequence was conducted with this vehicle using four iterations of the soot loading cycle, instead of only a single iteration.

Additional testing was performed with two Euro-3 diesel vehicles without a DPF, Diesel_H1, and Diesel_H2, used as the contamination source. These vehicles had elevated the PM background in the tunnel to levels that could be of interest in terms of characterizing a measurable contamination effect.

2.2 PM Sampling and Mass Measurements

A schematic of the laboratory setup is shown in the Abstract graph and Fig. 1. Two unique PM samplers were utilized in this study: a multi-filter PM sampler in a CVS system and a single filter PFD system. PM sampling was conducted following the procedures in CFR Part 1066.110 and associated references in CFR Part 1065, with the exception of changes to the protocol designed to test higher FFVs [24, 25].

CVS A multi-filter sampler was utilized that simultaneously collected PM with different gravimetric filter samplers from the dilute CVS. This PM sampler was built based on CFR part 1065 requirements, and it has a heated chamber to maintain an inside temperature at 47 ± 5 °C. Sampler A was used to collect a cumulative PM filter at different nominal FFVs during FTP or US06 tests. Sampler C was used to collect a Quartz filter for EC/OC samples. The CVS flow was set to provide a mean DF for phase 1 of the FTP and for the US06 of 7, based on the carbon balance calculation in CFR Part 1066.610. This provided average DFs from 8 to 12 for the other phases of the FTP, depending on the vehicle. These DFs are sufficiently high to prevent water condensation but allow for higher amounts of PM to be collected on the filter.

An AVL Micro Soot Sensor (MSS^{Plus}, Model 483 AVL Inc.) was used to sample directly from the CVS to provide real-time measurements of soot concentration. The AVL MSS^{Plus} exposes the sample stream to modulated laser light. The light absorbed by the particles produces pressure fluctuations related to the particle concentrations that are measured with a microphone. Additionally, a TSI Engine Exhaust Particle Sizer (EEPS, Model 3090, TSI Inc.) was used to sample directly from the CVS to provide real-time measurements of particle size distributions.

Total particle number (PN) was measured with a TSI 3776 ultrafine CPC. Solid particle number (SPN) was measured with a second TSI 3776 ultrafine CPC that was downstream of a catalytic stripper [26, 27]. The catalytic stripper used was 40 mm long with a diameter of 17 mm and was based on a cordierite monolith with a 400 cpsi cell density and a 6-mils substrate thickness. It had both oxidation and sulfur storage capability and was characterized according to the protocol outlined by Amanatidis et al. [27]. Both CPCs were connected to an ejector diluter (dilution ratio is constant at 8) that was used to collect samples from the CVS tunnel.

PFD The PFD used for this study was a commercially available, CFR Part 1065 compliant system (AVL Smart Sampler_478). The PFD was mounted upstream of the CVS and directly sampled from the raw exhaust. A PFD can help reduce interference from artifacts with higher PM mass collected on a filter by allowing lower DFs and eliminating the need for a long transfer line for the full exhaust, which can induce storage/release effects [14, 15]. The PFD used a sample flow proportional to the total exhaust flow, mimicking the varying dilution in the CVS as a function of exhaust flow rate. PFDs have been more prevalent for the measurement of emissions of large engines, since it becomes impractical to utilize a CVS for engines with very high exhaust volumes. In this study, the PFD extracted approximately 1 to 4% of the total exhaust volume, depending on the driving condition and FFV, and the CVS PM mass emission rates were corrected to account for the fraction of exhaust extracted by the PFD. The DF for FTP phase 1 was set to be 7 for the PFD.

Table 2 PFD conditioning and contamination test matrix[1]

Date	Test vehicle	Test cycle	Test duration (s)	Tunnel condition	PFD			EFM[2]
					FFV	DF	Unit[2]	
October/5/2016	n/a	NG Burner	2237	Cleaning #1	100	7	1, 2, and 3	1
October/6/2016	PFI_L	4 bag FTP	3344	Clean #1	100	7	1, 2, and 3	1
October/6/2016	PFI_L	US06 * 2	1192	Clean #1	100	7	1, 2, and 3	1
October/6/2016	GDI_H	Soot Loading	2242	Contaminating #1	100	7	1, 2, and 3	1
October/7/2016	PFI_L	4 bag FTP	3344	Dirty #1	100	7	1, 2, and 3	1
October/7/2016	PFI_L	US06 * 2	1192	Dirty #1	100	7	1, 2, and 3	1
October/7/2016	n/a	Tunnel Blank	3344	Dirty #1	100	7	1, 2, and 3	1
October/7/2016	n/a	NG Burner	3600	Cleaning #2	100	7	1, 2, and 3	1
October/8/2016	PFI_L	4 bag FTP	3344	Clean #2	100	7	1, 2, and 3	1
October/8/2016	PFI_L	US06 * 2	1192	Clean #2	100	7	1, 2, and 3	1
October/8/2016	GDI_H	Soot Loading	2242	Contaminating #2	100	7	1, 2, and 3	1
October/9/2016	PFI_L	4 bag FTP	3344	Dirty #2	100	7	1, 2, and 3	1
October/9/2016	PFI_L	US06 * 2	1192	Dirty #2	100	7	1, 2, and 3	1
October/14/2016	n/a	NG Burner	2282	Cleaning #3	100	7	1, 2, and 3	1
October/15/2016	PFI_L	4 bag FTP	3344	Clean #3	100	7	1, 2, and 3	1
October/15/2016	PFI_L	US06 * 2	1192	Clean #3	100	7	1, 2, and 3	1
October/15/2016	GDI_H	Soot Loading	2242	Contaminating #3	100	7	1, 2, and 3	1
October/15/2016	GDI_H	Soot Loading	2242	Contaminating #3	100	7	1, 2, and 3	1
October/15/2016	GDI_H	Soot Loading * 2	4484	Contaminating #3	100	7	1, 2, and 3	1
October/16/2016	PFI_L	4 bag FTP	3344	Dirty #3	100	7	1, 2, and 3	1
October/16/2016	PFI_L	US06 * 2	1192	Dirty #3	100	7	1, 2, and 3	1
October/16/2016	n/a	NG Burner	2080	Cleaning #4	100	7	1, 2, and 3	1
October/17/2016	PFI_L	4 bag FTP	3344	Clean #4	100	7	1, 2, and 3	1
October/17/2016	PFI_L	US06 * 2	1192	Clean #4	100	7	1, 2, and 3	1
October/17/2016	n/a	Tunnel Blank	3344	Clean #4	100	7	1, 2, and 3	1
10 days	Diesel_H1, Diesel_H2	3 bag FTP*5 and NEDC*4	18,285	Contaminating #4	100	7	1, 2, and 3	1
October/28/2016	n/a	Tunnel Blank	3344	Dirty #4	100	7	1, 2, and 3	1
November/22/2016	PFI_L	4 bag FTP	3344	Dirty #4	100	7	1, 2, and 3	1
November/22/2016	PFI_L	US06 * 2	1192	Dirty #4	100	7	1, 2, and 3	1
November/22/2016	n/a	NG Burner	8093	Cleaning #5	100	7	1, 2, and 3	1
November/23/2016	n/a	Tunnel Blank	3344	Clean #5	100	7	1, 2, and 3	1
November/28/2016	PFI_L	4 bag FTP	3344	Clean #5	100	7	1, 2, and 3	1
November/28/2016	PFI_L	US06 * 2	1192	Clean #5	100	7	1, 2, and 3	1

[1] The thermal conditioning tests were performed in triplicate

[2] Each PFD was used (1, 2, and 3) and only one EFM (EFM_A) to manage sample locations and transfer line length. The PM loading cycle is described in Sect. 2.3.2. The cleaning duration was 1 h of burner operation, but only 0.5 h of actual operation at the desired temperature. Real-time instruments collected PM data during testing, PM build up, and cleaning procedures

Gravimetric PM Mass Measurements Total PM mass samples for both the CVS and PFD were collected using Whatman 47-mm polytetrafluoroethylene (PTFE) filters. They were weighed using a CFR Part 1065-compliant microbalance with a neutralizer in a conditioned room meeting CFR Part 1065 requirements. Filters were weighed at least twice both pre- and post-test until two measurements within 3 μg were obtained. The stability of the weighing conditions was also monitored with 5 Teflon reference filters, which were weighed at least daily and sometimes hourly, which showed standard deviations of between 1.0 and 2.0 μg.

The PM emission results were background corrected based on average tunnel blank filter masses collected periodically over the course of the project, which included other separate studies that are discussed elsewhere [13]. Tunnel blanks were collected over a test with the same duration as the cumulative or individual FTP phases or US06 tests, but without exhaust flow. The average tunnel blank equaled to 10.5 ± 5.6 μg
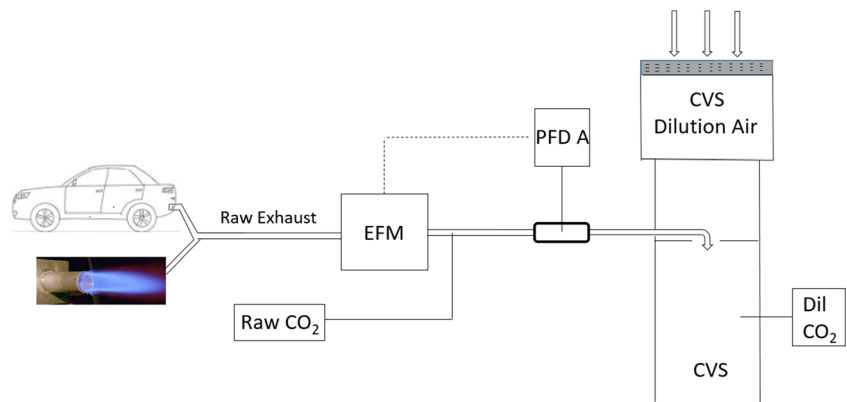
(average ± standard deviation, no. = 6) for the CVS probes and 3.1 ± 2.8 μg for the PFD sampler (no. = 6) based on the average tunnel blank values over the course of the full study. For this part of the study, four different tunnel blanks were collected, as indicated in Table 2 below.

2.3 Test Cycles and Study Design

The setup for this study is shown in Fig. 1. For vehicle testing, the sample system begins at the exit of the vehicle (i.e., the exhaust tailpipe) and continues until the gravimetric filter. The CVS and PFD shared the same exhaust transfer line from the vehicle's tailpipe exit. After the PFD sample probe splitter, the raw exhaust transfer line continued for another 3 feet before connecting to the CVS dilution tunnel. This is far shorter than the average transfer hose, which is allowed to extend to 10 m under part 1065.

A series of tests were performed with the tunnel in "clean" vs "contaminated" conditions. Table 2 shows the sequence that was used during the course of this study and the associated test conditions. The test sequence included initially cleaning the system, then "contaminating" the system by testing with GDI_H or Diesel_H, which allowed PM build up within the sampling system. In this study, contamination is defined as the deposition of PM and adsorption of semivolatiles onto the inner surfaces of the PM sample system that can be resuspended or desorbed from those surfaces on subsequent tests. Tests with PFI_L were then conducted after both the cleaning and "contamination" steps to evaluate impacts of testing with a contaminated vs a clean tunnel. A low PM emitting PFI vehicle was selected to evaluate these impacts since it was expected that contamination effects on a relative basis would have the most impact at low PM emission levels. A preparatory LA4 was run before the FTP tests with PFI_L for both the cleaning and contamination tests.

2.3.1 Cleaning Methodology

A natural gas (NG) burner was used to clean the tunnel. The NG burner was attached to the front end of the transfer line,

such that the cleaning included all elements of the sampling train, including the transfer line, exhaust transfer lines, and the CVS sampling system, as shown in Fig. 1. The NG burner is essentially particle free and provides sustained temperatures of ~ 400–450 °C at the entrance to the exhaust transfer line, with temperatures ranging from 150 to 300 °C measured at the PFD samplers and at the entrance to the CVS tunnel. The NG burner was utilized for periods ranging from 30 min to more than 2 h. Thirty minutes (or 1800 s) is the typical time needed to achieve low enough background levels in our other heavy-duty CVS tunnel, where the NG burner is typically used, for the measurement of vehicles equipped with diesel particle filters (DPFs). The cleaning cycle ended when the exhaust temperature, measured in the middle of the transfer line, reached 400 °C for the cleaning cycle 1, 3, and 4. For the cleaning cycle 2, a longer time was explored. The cycle went on from the time the entrance to the exhaust transfer line reached 400 °C until a decrease in particle number concentration was observed downstream of CVS to investigate whether even lower tunnel blank levels could be achieved. An even longer time was explored for the cleaning cycle #5, as the prior PM loading for this cycle was more than two orders of magnitude higher than the loading for all the other loading cycles. The CVS tunnel condition after using the NG burner was termed as clean in the present study. Real-time PM data and quartz filters for thermal optical analysis (TOA) for elemental carbon (EC) and organic carbon (OC) were also collected during the cleaning procedures to characterize the PM that was emitted during this process.

2.3.2 PM Loading Cycle

Several preliminary tests were evaluated to develop a cycle that was appropriate for loading the tunnel with soot. Figure 2 shows the cumulative and real-time PM-soot (MSS-483) that passed through the tunnel during the soot loading cycle that was selected for this study, which is called the Low Speed Soot Loading Cycle (LSSL), and associated soot PM for GDI_H. A total of 30 mg of PM-soot was emitted over the test cycle for GDI_H, while the temperatures in the transfer
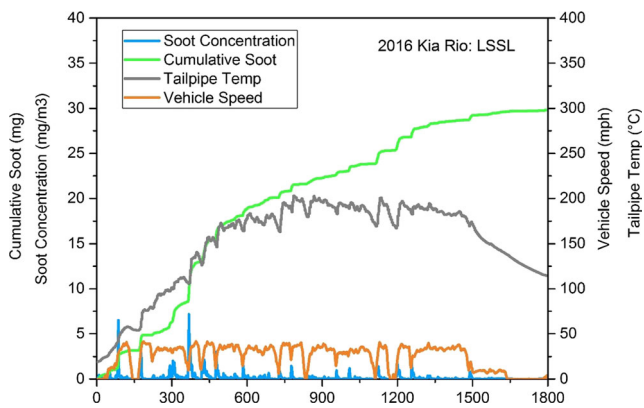
Fig. 2 Cold start soot loading test cycle showing cumulative soot loading

line were found to be below 200 °C. Maintaining temperatures below 200 °C was considered a priority in order to ensure that semi-volatile compounds would not desorb from the tunnel wall. These results suggest the proposed cycle provides sufficient levels of PM mass (30 mg) flowing through CVS without causing excessive transfer-line temperatures (< 200 °C). For the final soot loading cycle, an additional hill representing the most aggressive part of the LA92 cycle was added at the end of the cycle. During this hill, it was found that an additional 12 mg of PM was emitted, which would increase the soot exposure by another 40%. Although the temperature did exceed 200 °C during this hill, this only occurred for a short period near the end of the hill, and it was expected that limited desorption would occur under such conditions. The final soot loading cycle is presented in Fig. S1 in Supplemental Information.

The Diesel_H1 and Diesel_H2 vehicles, on the other hand, were not driven over the soot loading cycle. These vehicles were being tested over triplicate 3 bag FTPs and triplicate New European Driving Cycles (NEDCs) as part of another testing project. At the time of this testing, it was found that the tunnel blank levels had increased in the tunnel, so it was decided that an additional "contamination/cleaning" sequence should be conducted to see if these higher tunnel blank level contamination impacts would impact the PM emission rate of the PFI_L.

2.3.3 Contamination Characterization Testing

To evaluate the impact of the contaminated vs clean tunnel condition, PFI_L was tested over an FTP cycle and a US06 cycle with the sampling system in both a contaminated and clean state. Table 2 lists the test conditions and sequence. The FTP is the primary emission certification test for all LDVs in the USA, which includes a cold start phase 1, a stabilized driving phase 2, and a hot start phase 3 after the engine has been turned off for 10 min. The FTP can also be run as a 4 phase test by repeating the phase 2 stabilized driving cycle immediately after phase 3. The 4 bag version of the FTP

was used for this study, since the 4 bag FTP generally provides higher PM filter masses, which would make it easier to identify any contamination impacts. The US06 cycle is a single phase test that was incorporated into the regulatory procedures as part of the Supplemental Federal Test Procedure (SFTP) beginning in 2000 and is designed to represent more aggressive driving behavior. It should be noted that for the US06 cycle, the conditioning included the test FTP cycle and a pre-conditioning US06 cycle to help ensure repeatable emissions. That means the tunnel in either the clean or contaminated condition was contaminated/conditioned by a prior FTP and US06 prep cycle for the US06 test.

For this study, PM emissions were obtained using a single composite filter that collects PM cumulatively over the entire FTP. When a single cumulative filter is utilized as opposed to individual filters, the flow rates for the different phases must be adjusted to ensure that the cumulative filter provides the appropriate weighting between the different phases for the composite emissions factor, as described in CFR Part 1066.815(b). Thus, for the 4 phase FTP, the FFVs for phase 1 and 2 are adjusted to 75% of the nominal FFVs for phases 3 and 4.

3 Results

This section is organized into three main subsections: (1) PM loading tests, (2) PM cleaning tests, and (3) impact of prior loading and cleaning tests on the measurement of PM and PN emissions. The PM and PN emission impacts subsection includes three additional subsections covering tunnel blanks, PFD vs CVS comparisons, and PN results. The PM loading subsection describes the results of how much PM was emitted from the vehicles during the soot loading cycles. The PM cleaning subsection describes the amount of PM that desorbed during the cleaning of the transfer line and CVS and PFD samplers. The PM emissions impact section presents the PM emissions results for PFI_L following the conditioning of the sampling system into clean and "dirty" states. The tunnel blank impact subsection describes the impact of these clean and dirty states on tunnel blanks.

3.1 PM Loading Indicated by the Measurement with CVS and PFD

Assuming the amount of PM loading (or deposition) to the wall is proportional to the amount of PM passed through the system, PM loading was inferred by measuring the PM mass through the CVS and PFD sampling systems. The amount of PM emitted by the vehicle through the sampling tunnels during four soot loading cycles is provided in Table 3. Note that all of the PM in the exhaust goes through the CVS regardless of dilution. This is not the same for the PFD, as the PFD only samples a small fraction (1 to 4%) of the total vehicle exhaust.

Table 3 Total tailpipe emissions measured by the dilution systems

Soot loading cycle number	Vehicle	CVS_A (mg)	PFD_A (mg)	PM_Soot (mg)	THC (mg)
1	GDI_H	129.7	98.3	51.3	433.0
2	GDI_H	35.8	36.3	28.8	285.5
3	GDI_H	113.6	104.7	81.4	1326.1
4	Diesel H1 and H2	8565.6	n/a	n/a	n/a

Data in the table are based on total mass (mg) passing through the CVS system. Columns PM_soot (measured by MSS) and THC pertain to the CVS, but only 1–4% of emissions measured by PFD_A, which accounts for dilution rates of the PFD_A, pass through the PFD

The PFD and CVS should agree on the vehicle's PM emission rate, however, as the amount of exhaust that goes into the PFD system is proportional to the exhaust flow rate. For the first contamination cycle, the GDI_H vehicle emitted up to 10 mg/mi (CVS_A) for the soot loading cycle, which amounted to 129.7 mg of PM mass passing through the CVS sampling system. The PFD system showed a lower amount of mass emitted by the GDI_H vehicle (98.3 mg, 25% less mg than CVS_A) for the first cycle for unknown reasons. The PFD and CVS showed more similar levels of PM mass emissions for the subsequent second and third cycles (> 90% agreement). The second soot loading cycle provided ~ 35.8 mg (3 mg/mi) of PM, which is 72% less than the first loading cycle passed through the sampling system. To provide greater soot-loading, the cycle was repeated four times for the soot loading test #3, which provided 113.6 mg of PM passing through the CVS system. For each of these soot loading cycles, the impact of the PM build up in the CVS on the PM emission rate of the PFI_L was minimal, however, as discussed below.

Since the tunnel loadings provided by GDI_H did not seem to have a significant impact on the PFI-L emissions, a higher PM emission source was sought. Two additional non-DPF equipped diesel vehicles were selected from a different UCR testing program for this purpose. These vehicles had average FTP emission rates of ~ 50 mg/mi. These vehicles were tested for 11 days as part of this other program, where approximately 8565 mg of PM passed through the raw exhaust transfer line and the CVS PM sampling system (note the PFDs were not operated during this testing). The results of this contamination cycle are denoted as soot loading test #4.

3.2 PM Cleaning

During the cleaning stage with the natural gas burner, the PM mass, particle size distribution (PSD), and PM-soot were measured. Table 4 shows the integrated PM mass and PM-soot collected during the cleaning stage, as well as the time the NG burner was used for each cleaning run. For CVS_A, the total PM mass removal was very similar for cleaning tests 1 and 3 (30.0 mg vs 33.9 mg removed), and tests 2 and 4 (7.1 mg vs 9.9 mg), but did not show consistent trends over the full sequence of tests after the GDI_H loading. The PFDs also showed inconsistent trends with respect to PM removal after the GDI_H loading. For PFD_A, the lowest PM mass removal measured was during the first cleaning test. PM removal following subsequent loading cycles with GDI_H was higher but varied from test-to-test. PM removal was also not found to be a strong function of NG burner time, as the amount of PM removed for the 1 h burner was lower for the CVS and near the average levels for the PFD compared to the 30 min burn times. This indicates that the excessive heat loading in the transfer line and moderate heat loading in the CVS and PFD by the NG burner in the 30 min burn times had already desorbed a majority of volatile and semi-volatile materials from the surface. It also removed some soot particles. Also in the longer burn, the combination of high temperatures and longer time may also have led to desorption of semi-volatiles from the collection filters.

Further insight can be obtained by calculating what percent of mass is removed during the cleaning process compared to the amount of PM mass that passes through the tunnel during the previous loading cycle. This percentage is also included in Table 4. The percentage of soot removed as a function of the PM mass from the loading cycle was relatively low, ranging from 0.1 to 0.4%. This is similar to a value of 0.14% that was also calculated by Kittelson and Johnson [28] for the total loss of particles at a 0.3 μm diameter, a typical mass mean diameter, in a heavy-duty diesel sampling CVS system during the Heavy-Duty Transient Test. Resuspended soot particles also tend to be coarse mode particles, which may be large enough to be removed by the PM2.5 cyclone installed in the sampling system. Another consideration is that the steady flow conditions for the burner test may have been less likely to re-entrain soot than a transient engine test. The very low percentage of soot removed by cleaning reflects these aspects. On the other hand, the percentage of total PM removed during cleaning cycle ranged from 0.7 to 93.8% for CVS measurement and 16.2 to 104.7% for PFD measurement. These higher particle release percentages are not explainable by particle resuspension alone, particularly relative to the previous loading cycle. This suggests that much of the PM removed during cleaning is semi-volatile and that soot plays a minor role, either because it

Table 4 Cleaning cycle results

Cleaning	PM removed by cleaning (mg)			Burning time (s)
	CVS_A (mg)	PFD_A (mg)	PM_Soot (mg)	
1	29.8	0.083–0.332	0.4	2237
2	7.1 (5.5%)	0.209–0.836 (21.3%)	0.2 (0.4%)	3600
3	33.6 (93.8%)	0.380–1.520 (104.7%)	0.1 (0.3%)	2282
4	9.9 (8.7%)	0.170–0.680 (16.2%)	0.1 (0.1%)	2080
5	60.1 (0.7%)	118.1	2.1	8093

In parenthesis is the percentage of the PM mass removed during cleaning compared to the PM mass that passed through the tunnel in the previous loading cycle. Columns PM_soot and THC pertain to the CVS. Note the values for PFD_A has a range as the amount passing through the PFD was estimated at 1–4% of the difference in emissions. Note for cleaning cycles 4 and 5, the exhaust from the immediate prior loading cycle did not pass through PFD because the extent of loading could potentially have damaged the PFD

is not a major contaminant, or because it is only released in the form of large, > 2.5 μm size, particles. The wide range of PM mass release fractions also indicates that PM build up accumulates over a longer period of time than can be simulated with a few tests with a vehicle emitting at the 3–10 mg/mi level.

The amount of PM removal during the fifth cleaning test following the loading with Diesel_H1 and H2 did increase relative to the amount of PM removed following the GDI_H cleaning tests for both the CVS_A and PFD_A measurements. Total CVS_A PM mass was approximately 2 to 6 times higher for the cleaning stage after the diesel vehicles, while the total PM mass for PFD_A was approximately 3 to 14 times higher. Although significantly more PM was removed following the cleaning test following the diesel loading, the level of increase in the PM removal was still considerably less than the 66 to 237 times greater PM mass that passed through the dilution tunnel during the diesel loading compared to the loading with GDI_H. This indicates that the amount of PM build up in the tunnel does show some dependence on the amount of PM that passes through the tunnel, but that there appears to be a saturation limit as to how much PM (soot + organics) is adsorbed onto the surfaces of the sampling system.

Only a small fraction of the PM removed was soot, as shown in Table 4. Quartz filters collected during the cleaning cycles also showed that 90% or more of the total carbon was OC, with EC levels comparable to those measured with the MSS. PM-soot showed more consistent trends, with the amount of PM removal decreasing in going from 0.43 mg for the cleaning cycle #1 to 0.005 mg for cleaning cycle #4 after the GDI_H loading. The highest amount of soot removed was also found after the loading with the diesel vehicles, which varied from 5 to 42 times the PM removed compared to the loading cycles done with GDI_H.

The real-time number weighted PSDs measured by an EEPS, as shown in Fig. 3, showed relatively higher PN concentrations for the first cleaning cycle compared to the second cycle (~ 10 times lower concentration). It is clear from Fig. 3

that nearly all of the PN from cleaning cycles is in the nucleation mode size range between about 5 and 30 nm. Particles in the size range usually comprise most of the particle number and are formed by nucleation of semi-volatile materials. Soot particles are typically found in the accumulation mode size range between about 30 and 300 nm. The lack of material in this size range is consistent with the very low soot concentration.

3.3 Impact of Prior Loading/Cleaning Tests on the Measurement of PM and PN Emissions

The CVS PM, PFD PM, and PM-soot emission rates for testing of PFI_L with the sampling system in both dirty and clean states are presented in Fig. 4a and b for the FTP and US06 tests, respectively. The blue arrows in the figure show the cleaning and/or soot loading events that were conducted before each vehicle emission test. Emission tests that occurred after soot loading tests are defined as dirty tests, and emission tests that occurred after cleaning cycles are defined as clean tests. The text added next to the top end of the blue arrows in the figure shows the amount of mass (mg) removed from the CVS_A sample system during cleaning cycles or the amount of mass that passed through the CVS_A sample system during the soot loading cycles. For example, the amount of mass removed from the sample system was 30 mg during the first cleaning cycle (CVS_A probe) and the amount of mass that passed through the sample system was (130 mg) for the first soot loading event.

Overall, the PM test results did not appear to show any consistent trends with respect to cleaning and loading conditions. PM did not consistently decrease after cleaning cycles or increase after loading cycles. FTP PM emissions rates of PFI_L were highest for the test immediately after the initial cleaning cycle and for the tests conducted after the soot loading cycle with the diesel vehicles, see Fig. 4a and b. The FTP PM and PM-soot emissions for the initial clean/dirty cycle on GDI_H appeared to stabilize, with the exception of slightly
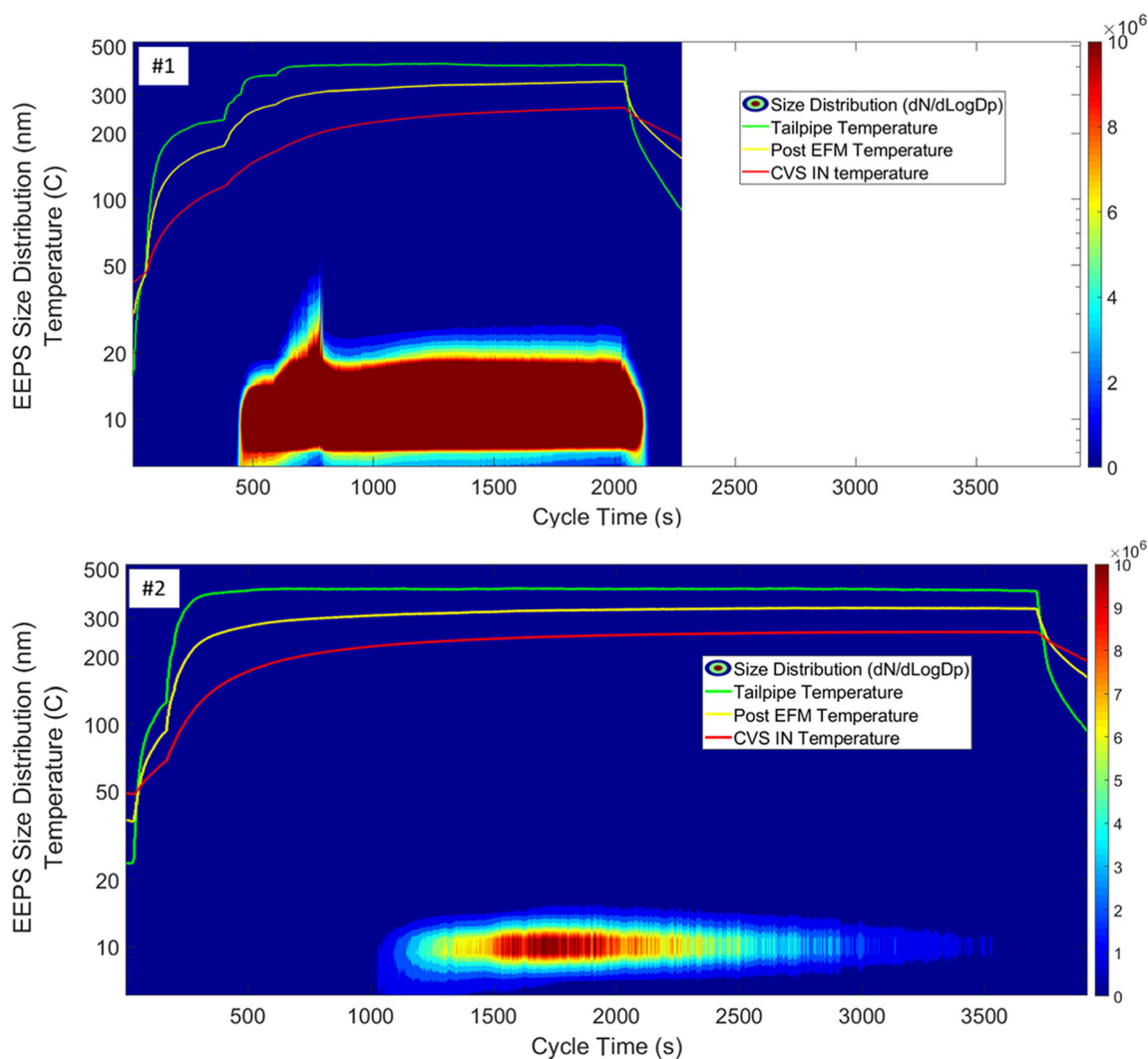
Fig. 3 Real-time EEPS data for the burnout response during #1 and #2 cleaning steps

higher values for clean test #3. After PM loading with the diesel vehicles, the FTP emission rates appeared to increase from 0.08 to 0.25 mg/mi. The increased amount of emission rates must be due to PM released from the sampling system. On the other hand, cleaning did not seem to decrease the emission rates. In fact, emissions were higher immediately after cleaning 1, 3, and 5, reduced after cleaning 2, and about the same after 4.

The first US06 test also showed the highest PM emission rates. The US06 test appeared to stabilize over the first four test sequences (#1 clean/dirty and #2 clean/dirty). PM emission rates from US06 cycle are known to stabilize to smaller values after three or four initial tests [10]. These results are also consistent with the FTP test results. PM emission rates increased to a higher level again for the 3rd clean test and then decreased for the following test (the 3rd dirty test). It should be noted that the US06 tests were all preceded by the FTP tests and a preconditioning US06 cycle. As such, initial sampling system conditions for the US06 tests are somewhat different

than those for the FTP, which were conducted immediately after the cleaning or soot loading cycles. Nevertheless, it is interesting to note this down-up-down trend is the same over the #1–3 (clean/dirty) tests for both the FTP and US06 tests. Close examination shows a similar impact of cleaning on PM mass emissions to that in the FTP tests with higher emissions after cleaning in tests 1, 3, and 4, reduced emissions for 2, and slightly reduced emissions for 5. This down-up-down trend was not observed for PM soot, which stabilized for the FTP and trended downward US06 tests after the initially high PM-soot emissions, until it spiked (or de-stabilized) after PM loading with the diesel vehicles.

Since a variety of factors could potentially contribute to variability between different tests at these low levels, additional analyses were done where the CVS and PFD PM mass measurements were rescaled such that the soot component equaled the average value of 0.057 mg/mi, such that the variations in total PM mass could be evaluated under conditions where the PM soot mass was equalized. These results are

Fig. 4 a CVS, PFD, and PM-soot emission rates for clean/contamination for the FTP. b CVS, PFD, and PM-soot emission rates for clean/contamination for the US06
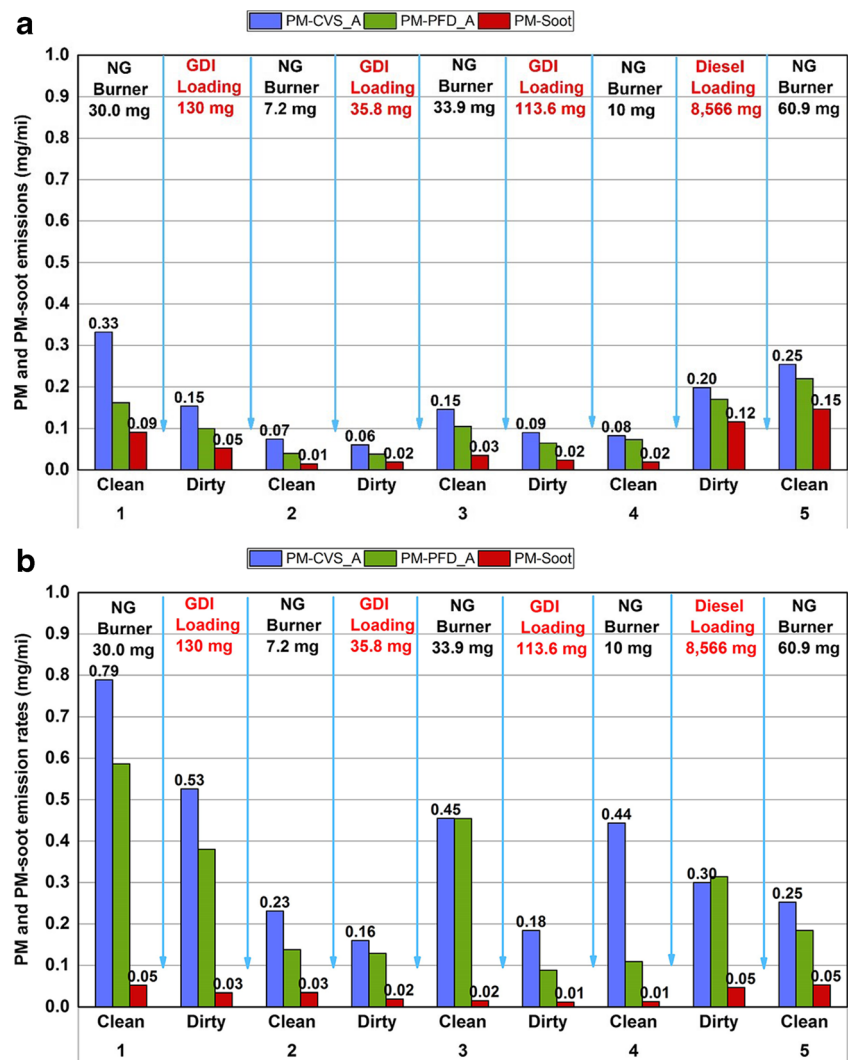
presented below in Fig. 5. Examining the results from this perspective revealed some additional trends of interest. In particular, it was found that the rescaled PM-CVS_A values were higher for each clean emission test compared to either the previous or next dirty emission test. Similarly, the rescaled PM-CVS_A decreased for each dirty emission test compared to the previous or the next clean test.

Although the rescaled FTP results appear to be counterintuitive at a first glance, they can be understood as follows. While the cleaning procedure removes both deposited soot and organics, it also reduces the available surface area for organics to condense on. Soot loading cycles deposit soot and organics on the wall. This could be why lower PM emissions were observed after loading, and higher PM emissions were observed after cleaning in the normalized plot (Fig. 5).

Since the US06 is a more aggressive cycle, the associated exhaust temperatures could lead to greater desorption of PM from the sampling system surfaces. The US06 PM results were plotted against maximum exhaust temperature during the US06 cycle in Fig. S2. The differences in maximum

temperature were within 13 °C between the different US06 tests, however, and there was not a strong correlation between CVS PM and PFD PM and maximum exhaust temperature for the US06. On the other hand, maximum exhaust temperature showed some correlation with MSS measured soot with $R^2 = 0.40$. The US06 cycle is known to produce large test-to-test emissions variations even when rigorous prep cycles like the one used in this study are adopted [20, 21]. As the US06 cycle was done after an FTP test and US06 prep cycle, such that the actual US06 cycle was not conducted immediately after the cleaning/loading cycle, the variations in CVS PM and PFD PM could be due to factors other than the cleaning/loading procedure itself. The consistency between FTP and US06 tests for a given sequence does suggest that tunnel conditions have an impact on the results, however. Further investigation is needed to better understand these variations.

The trend toward stabilization between cleaning cycles suggests that an alternative approach to obtaining stable and representative emissions would be to stabilize the tunnel to the vehicle, that is to conduct several stabilization tests with the

Fig. 5 Normalized CVS, PFD, and PM-soot emission rates to remove test-to-test variation for clean/contamination, FTP cycles
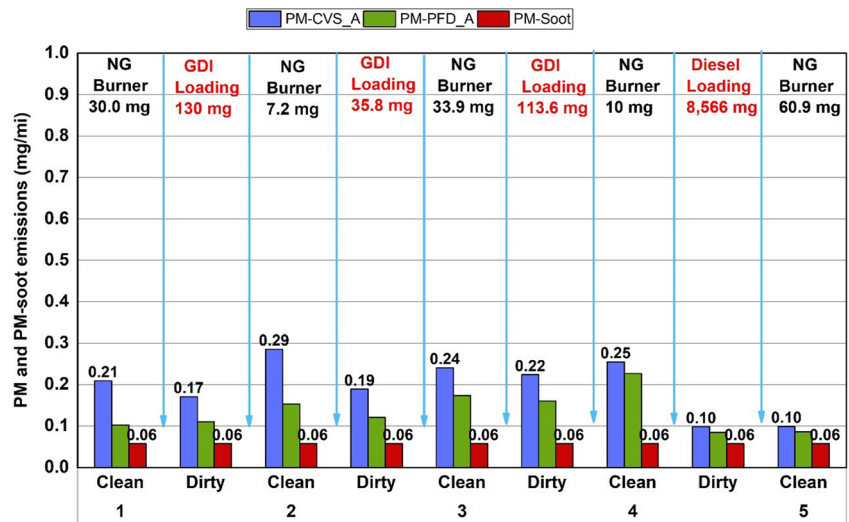
vehicle to be tested, or a similar vehicle. Kittelson and Johnson [28] suggested that an approach to improve repeatability in heavy-duty emission tests would be to equilibrate the tunnel to the engine, which is to run the engine through its load range to condition the sampling and dilution system to the engine.

Tunnel Blanks The CVS and PFD tunnel blanks associated with this testing are shown in Table 5. The CVS TBs measured during the contamination test show reduced TB mass after cleaning and increased TB mass after soot-loading cycles with GDI_H. TBs for the PFD were lower than those for the CVS in general, with the exception of the 10/17/2016 TB for PFD_A. After the high emitting (averaging ~ 50 mg/mi) diesel vehicles (Diesel_H1 and Diesel_H2) were run, where over 8000 mg of PM passed through the sample system (PFDs were connected, but did not sample during this testing), the CVS TB decreased to 3 μg after the soot-loading cycle and increased to 16 μg after the cleaning cycle. This phenomenon is exactly opposite to what was observed when GDI_H was used for the soot loading cycle, where the TB was found to decrease after a cleaning cycle. We speculate that is because desorption from the CVS depended on the available surface area for OC adsorption. It is likely this available surface area increased after the relatively large loading of soot by

Diesel_H1 and Diesel_H2 prior to decreasing after the last cleaning cycle, as discussed further below.

PFD vs CVS Comparison The PFD showed lower overall PM mass emissions compared to the CVS for the FTP and US06 tests. PFD_A's PM emissions were 30% and 29% lower on average compared to CVS_A for the FTP and US06, respectively. However, these differences are relatively small on an absolute scale. The results of the direct PFD comparisons showed an absolute difference of − 0.046 mg/mi vs − 0.12 mg/mi for the FTP and − 0.11 mg/mi vs − 0.17 mg/mi for the US06. These absolute differences were actually less than those found during a more systematic evaluation of PFD and CVS differences that was conducted in conjunction with this study and is presented elsewhere [13]. Additionally, the PM emissions comparison did not seem to improve as the cleaning steps were performed, suggesting a bias between CVS and PFD remained even though the tunnel blank for the CVS decreased (15 to 3 μg) at cycle #3, as discussed in greater detail in the next section.

PN and SPN Results PN and SPN emissions for the FTP and US06 are shown in Fig. 6a and b, respectively. Over the FTP test, PN and SPN were highest for the tests conducted in conjunction with the highest PM loading with the diesel

Table 5 Tunnel blank (mg) results for all PM systems utilized for samples collected throughout the study

Date	Task	Test	CVS_A	PFD_A	Tunnel Condition
August/31/2016	Pretest	1	0.0090	0.0010	Dirty
September/26/2016	Pretest	2	0.0150	0.0050	Dirty
October/7/2016	Test	1	0.0150	0.0033	Dirty (after dirty cycle 1)
October/17/2016	Test	2	0.0050	0.0066	Clean (after clean cycle 4)
October/28/2016	Test	3	0.0030	− 0.0011	Dirty (after dirty cycle 4)
November/23/2016	Test	4	0.0160	0.0037	Clean (after clean cycle 5)
Value used (mg)			0.0105	0.0031	

Fig. 6 **a** PN and SPN emission rates for clean/contamination FTP cycles. **b** PN and SPN emission rates for clean/contamination US06 cycles. Black text is the NG burner mass collected during the cleaning step and the red text is the amount of PM emitted by the contamination vehicle as measured by the CVS_A probe. Results from PFD_B were similar
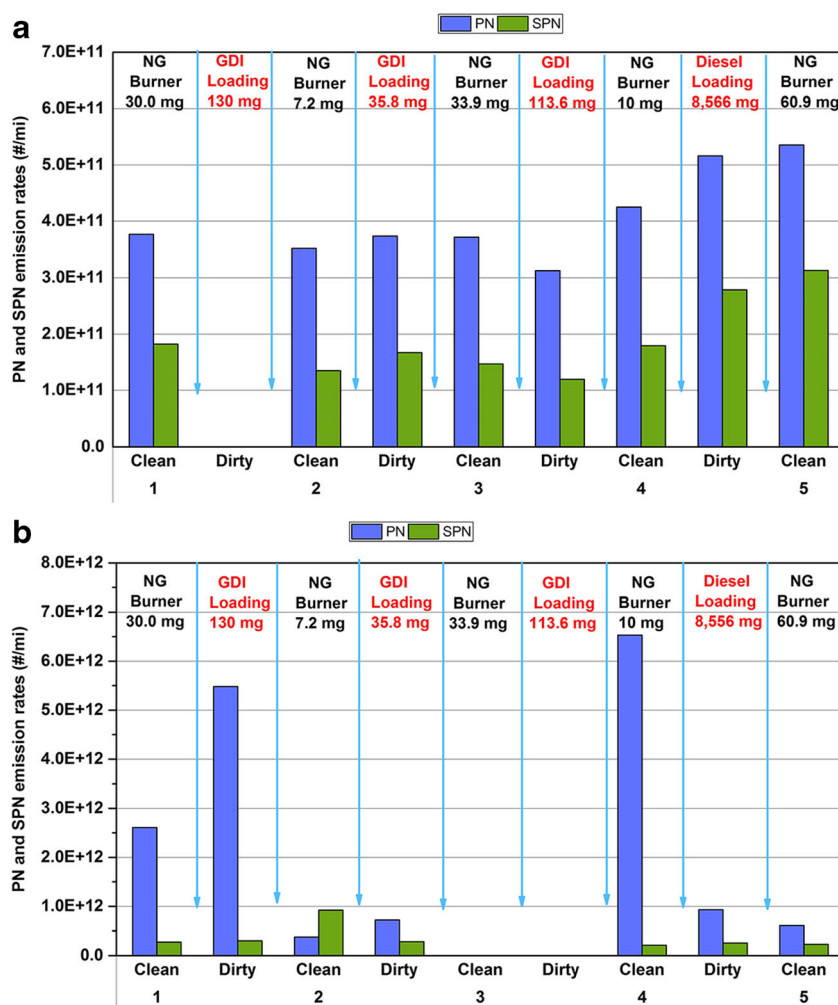

vehicles and showed similar levels after the NG burner cleaning cycle. The PN and SPN emissions for the FTP tests were lower for the contamination tests done in conjunction with the GDI_H vehicle and did not show any strong trends during those tests. Somewhat surprisingly, both PN and SPN results showed less test-to-test variation than the PM tests. This suggests that these suspended particle number measurements are less influenced by desorption/adsorption from the loading and cleaning cycles than gravimetric measurements.

The US06 results showed very different trends for PN and SPN emissions. The ratio of PN to SPN was much higher than for the FTP, indicating that most of the particles are mainly semi-volatile, which are removed by the catalytic stripper. This is consistent the relatively small fraction of measured PM soot, as seen in Fig. 4b, and with previous studies that have shown the particle measured during US06 tests are predominantly composted of organic carbon [20, 21]. Interestingly, PN emissions for the first clean and dirty and fourth clean US06 tests with the GDI-H vehicle were the highest, and PN emissions for the diesel contamination tests were actually lower than those for many of the GDI_H contamination tests. This differs from the

trends seen for the FTP tests, but it is consistent with the hypothesis that soot loaded walls scavenge semi-volatile materials and suppressing nucleation. SPN did not show any consistent trends between the diesel and GDI_H contamination tests or between the dirty and clean tests. The nature of the higher PN emissions rates for some of the GDI_H contamination tests is not readily apparent. Examination of the real-time PN emissions for the individual US06 tests showed that the higher PN emissions were found over the full duration of the test and do not simply represent a single large spike that occurred during the testing. The temperature profiles between the different US06 tests were also similar, however, so differences in the release of PM from the CVS/sampling system due to heat should be small. Further study is needed to better understand factors that can lead to PN emissions differences between tests.

Additional analyses were also conducted to evaluate the correlation between the total PM mass as measured on the gravimetric filters, and the MSS and SPN measurements. These correlations are shown in Fig. 7a–c for the FTP and Fig. 7d–f for the US06 cycles. Solid markers represent results from contamination tests and empty markers represent those
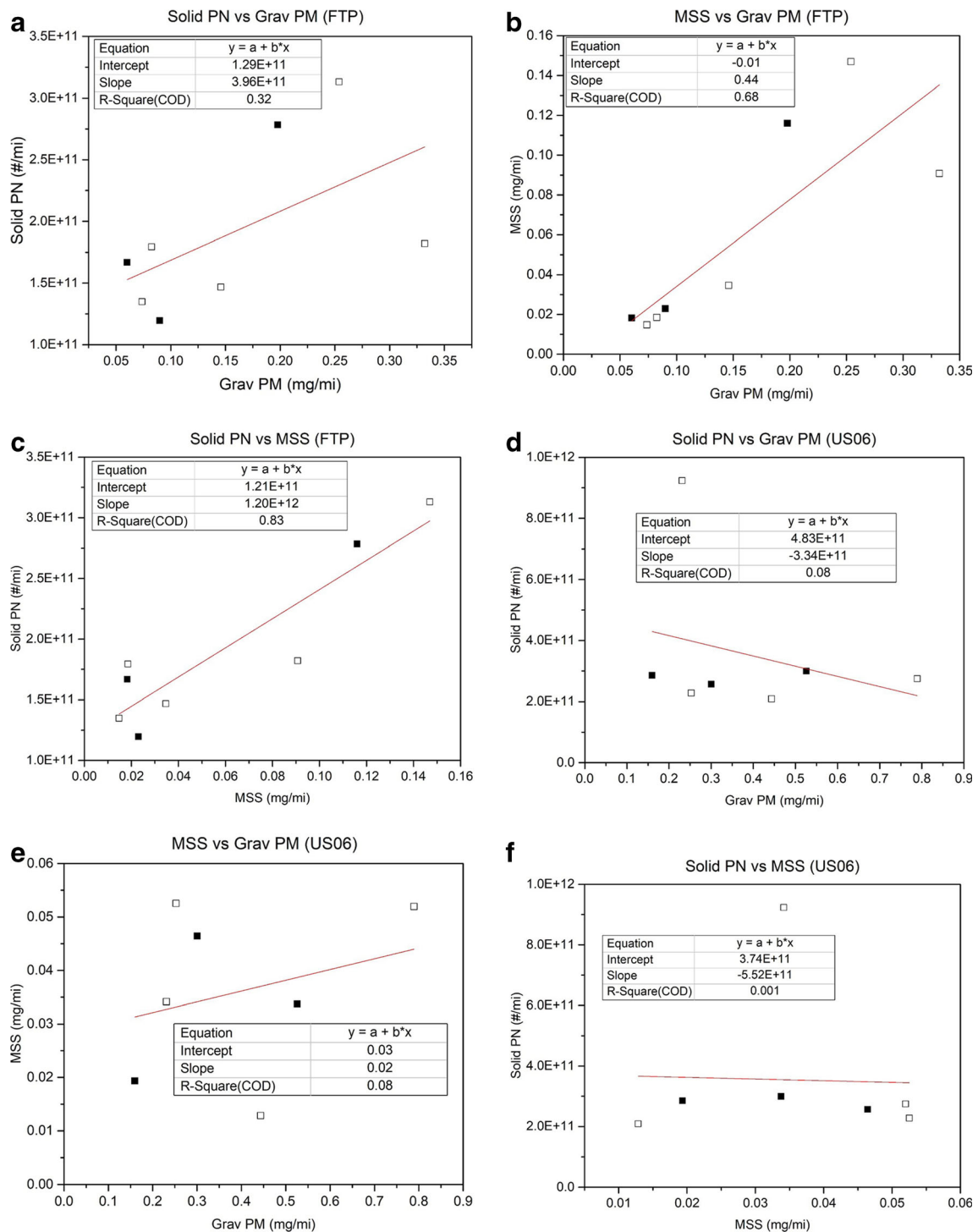
Fig. 7 Correlations between SPN, MSS, and gravimetric PM. Open symbols are tests after the cleaning cycle; solid symbols are tests after the loading cycle. **a**–**c** are for FTP cycle and **d**–**f** are for US06 cycle

from clean tests. No separate trends were found between contamination tests and clean tests. The FTP shows a positive correlation between the MSS and gravimetric PM ($R^2 = 0.68$) and the MSS and SPN ($R^2 = 0.83$), with a weaker correlation between the gravimetric PM and SPN ($R^2 = 0.32$). Over the US06 test, however, the correlation between the

MSS, SPN, and gravimetric PM mass measurements was poor. It is not surprising that neither the SPN nor MSS correlate well with gravimetric PM mass because PM mass is mainly semi-volatile for the US06 test. On the other hand, one might expect a better correlation between SPN emissions and MSS mass, i.e., solid number vs solid mass, but the plot

shows relatively constant SPN emissions independent of MSS mass. A possible, but not very convincing, explanation is that smaller particles, which constitute most of the particle number, are less influenced by test-to-test variability than larger particles that constitute most of the particle mass. Clearly, more work is needed to understand this relationship.

4 Summary and Conclusions

To evaluate the impacts of contamination and possible differences between the CVS and the PFD, a series of tests were performed with the tunnel in a clean vs contaminated condition. A GDI vehicle (10 mg/mi) and two diesel vehicles without DPFs (20–80 mg/mi) were used as a source of contamination for the tunnel. A soot loading cycle, called the Low Speed Soot Loading Cycle (LSSL), was successfully developed and utilized to pass 36 to 8600 mg of PM mass through the CVS tunnel. The tunnel was then "cleaned" using a natural gas burner that produced sustained heat (up to 400 °C for 60 min), and the nature of the desorbed PM was studied. The impacts of the preceding contamination and cleaning tests on the following quantification of PM emissions of a low PM-emitting PFI vehicle were also evaluated.

The results from cleaning desorption tests indicated that the majority of particles released during the high-temperature cleaning tests were semi-volatile in nature with little presence of soot. There was some increase in the amount of soot re-suspending from the tunnel, however, after a relatively high level exposure to exhaust from a diesel vehicle with a DPF. Combining the results of the contamination tests coupled with the associated cleaning tests, it was found that the amount of PM buildup on the surface of a typical dilution tunnel is a complicated function of the amount of exposure to PM, the longer term history of PM accumulation, and also potentially the type of vehicle exposure. Our results showed that PM buildup accumulates over a longer period of time than can be simulated with a few tests with a vehicle emitting PM around the 3–10 mg/mi level. Evaluations done with higher emitting diesel vehicles showed that the amount of PM buildup in the dilution tunnel does show some dependence on the amount of PM that passes through the tunnel, but that there appears to be a saturation limit as to how much PM is adsorbed onto the surface of the sampling system.

At first look, it appeared that there were no consistent trends with respect to the impacts of dirty and clean tunnel conditions on PM emission measurements from the low PM-emitting PFI vehicle. In particular, PM did not consistently decrease after cleaning cycles or increase after contamination cycles. Upon closer examination, however, we found emission results between FTP and US06 tests are somewhat related. Specifically, when a FTP test had a high emission rate so did the following US06 test and vice versa. This is likely because of similarities in the wall conditions in terms of desorbing or adsorbing. An intriguing trend was also found when the CVS and PFD PM mass measurements were rescaled such that the soot component equaled the average value of 0.057 mg/mi for the FTP results to remove the effects of test-to-test variation. The rescaled PM values were consistently higher for each clean emission test compared to either the previous or next dirty emission test. Some trends of lower PN emissions following higher PM loads with diesel vehicles were also seen. These somewhat counterintuitive findings could be due to contamination and cleaning tests decreasing and increasing, respectively, the amount of available surface area for condensation of organics, which could have an impact on the dilution tunnel adsorption/desorption characteristics. In particular, if the surface area is reduced through cleaning, then a smaller fraction of PM may condense on the PM sampling system and dilution tunnel, leading to higher measured PM levels. On the other hand if the surface area is increased through contamination, then a larger fraction of PM may condense on the PM sampling system and dilution tunnel, leading to lower measured PM levels.

The test-to-test variation in PM mass associated with contamination was in the range of 0.2 mg/mi for the FTP and 0.5 mg/mi for US06. These are less than, but not negligible compared to the proposed 2025 CA standard of 1 mg/mi. Further, although solid particle number is not currently regulated in the USA, the variability in SPN observed in these tests, 2×10^{11} and 5×10^{11} #/mi for FTP and US06, respectively, was not negligible compared to the current EU standard of 6×10^{11} #/km (9.7×10^{11} #/mi). While there has been a lot of discussion on this topic, there is lack of data on this issue. The study was the first experimentally designed investigation on the issue of contamination in vehicle PM emission measurements. This phenomenon is complex due to the interplay between the sampling system, emissions from the test vehicle, and history effects from the previous tests. The current study provides a framework for future studies to better understand this complicated phenomenon. Heat and mass transfer in the exhaust and sampling system clearly play an important role, and further work combining modeling of these processes with further experimental measurements is indicated.

Compliance with Ethical Standards The authors declare that they have no competing interests.

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